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CHEMICAL

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LIFTING THE 'CURTAIN'

SHORTCOMINGS in the chemical industries of some of the Soviet bloc countries have been the subject of comment in the press of those countries recently. For the first time production figures are being given, where in the past only percentage increases or decreases in production have been available.

This is a welcome change, for it gives the chemical industries of western countries for the first time an indication of capacities. On that basis a notified percentage increase begins to mean something. In attempting to find a reason for this change in policy, it is perhaps significant to recall that the Soviet Prime Minister has in the past few months more than once criticised the Soviet chemical and plastics industries. This might well have influenced those responsible for these industries in the satellite states.

At any rate, the current issue of *Polish Foreign Trade* puts it on record that: 'Placed on the one hand alongside the development of the chemical industries in other countries and on the other alongside the rising home demand and the great possibilities for expanding industry, the rate of development of the Polish chemical industry cannot escape criticism'.

While this particular journal says little more by way of criticism, details of the final stage of the 1955-60 five-year plan show that despite large-scale investments aimed at forcing exports of finished chemical goods and stocking the home market with a view to reducing imports, targets have not been reached and that the import-export programme will continue unchanged. Imports of raw materials for the artificial fabric and leather industries have increased, as have intakes of rubber. Imports of agricultural chemicals, such as fertilisers and raw materials for chemical fertilisers have risen. The report on the plan says that Polish capacity for such products must be increased.

It is stated that under the current plan, the output of the Polish chemical industry will by 1960 be more than double the 1955 figure. Sectors of the industry in which continued expansion is projected by the Polish Government are synthetic organic production via the processing of acetylene and coal-tar semi-products. A number of new coal-tar products are now being produced in Poland for the first time and these include methanol, phenol and acetic acid. It is officially anticipated that the organic synthesis industry will develop more rapidly than any other branch of chemical production and will supply material necessary for the further development of the synthetic fibre industry as well as for plastics, synthetic rubber and synthetic washing agents.

The fertiliser industry is also to be further expanded to enable the demand of Polish agriculture to be fully met. Production of nitrogenous fertilisers is expected to reach, by 1960, almost 300,000 tons, against 175,000 in 1956. Output of phosphorus fertilisers is to rise over the same period from 129,000 to 229,000 tons. A new and rapidly developing branch of the industry is that of insecticides, fungicides, herbicides, etc.

Output of pharmaceuticals is expected to increase by 150 per cent, reaching by 1960 the figure of Zl. 1,881 million, with the accent on antibiotics, vitamins, TB preventatives and pharmaceuticals for the treatment of cardial diseases.

The following is the official table showing output of the Polish chemical industry in the years 1949, 1955 and 1957, in '000 tons, unless otherwise stated.

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			1949	1955	1957
Sulphuric acid (100 per cen	t)		275.8	450.0	498.8
Calcined soda (98 per cent)			120.6	210.8	232.2
Caustic soda (96-98 per cen	t)	***	56.4	101.6	135.4
Synthetic ammonia		***	48.8	166.5	236.9
Carbide (75 per cent)			153.9	211.3	210.0
Gaseous chlorine			5.0	8.1	27.4
Nitrogen fertilisers, N			73.9	154.1	200.6
Phosphoric fertilisers, P.O.		***	131.9	123.5	138.3
Synthetic methanol	***	***	0.4	8.8	15.1
Synthetic phenol			_	3.5	4.7
Synthetic acetic acid			-	3.2	7.6
Dyestuffs			3.8	6.6	7.5
Enamels		***	10.7	48.3	64.8
Penicillin (in mld of units)			Chronic	6,238.8	9,452.1
Insecticides, weedkillers, fu			2.5	19.1	23.6
D 1 1 C C	-				The A.

Production figures from the Czech Ministry of Trade indicate that while many branches of chemical manufacture in Czechoslovakia showed an increase for the first half of 1958 compared with the same period of last year, in some cases production did not come up to expectation.

The largest increase was in the field of synthetic motor fuel, production of which went up by 22 per cent on figures for the first six months of 1957 to reach a total for the period of 173,000 tons. Figures for nitrogen fertilisers rose by 20 per cent on last year to a total of 46,000 tons. Phosphate

fertilisers went up by only 3 per cent, less than had been expected, to reach the figure of 57,000 tons for the half-year.

An account of the failure of East German industry to meet chemical production targets has been issued in Berlin. It states that the chemicals under-produced in the first six months of this year were sulphuric acid, calcium carbide and calcinated alumina. Sulphuric acid figures for the half-yearly period, however, at 263,000 tons, were an increase on the 258,000 tons produced in the corresponding period of 1957. Other chemicals, too, says the report, which were under-produced this year were nevertheless manufactured in greater quantity now than they had been last year.

There can be little doubt that the drive to raise the capacities of basic chemicals in Soviet bloc countries will eventually lead to higher outputs of many other chemicals and of synthetic fibres, plastics, and synthetic rubber. The countries concerned are likely, on a short-term basis, to prove fruitful export outlets for the UK chemical industry. They have, however, proved difficult areas with which to trade and firms interested in business with the Soviet countries would be well advised to make regular visits to gain first-hand knowledge of the potential markets, trading methods, financial arrangements.

POLYMERISATION IN AQUEOUS SYSTEMS

RESEARCH has thrown much light on the mechanism and site of polymerisation in aqueous emulsion systems where the dispersing agents are relatively simple soaps. Little is known, however, about polymerisation in aqueous systems containing protective colloids such as gelatin, i.e. the so-called suspension, bead or pearl polymerisation.

This lack of information has been pointed out by two Russian workers (A. S. Shevlyakov and K. S. Minsker, Kolloid Zhur., 1958, 20, 23).

From the overall kinetics of the process it has been assumed that polymerisation takes place mainly within the dispersed monomer droplets by a mechanism identical with that of bulk polymerisation. For this reason the process is sometimes called micro-bulk polymerisation. From their results Shevlyakov and Minsker conclude that this assumption is not justified since they show that a great deal of polymer formation also takes place in aqueous solution. The relative importance of the two sites of polymerisation is controlled mainly by the nature of the initiator.

The method of investigation consisted in polymerising vinyl monomers, coloured with a water-insoluble dye, in the presence of a number of initiators and colloids, with subsequent examination of the polymer particles formed. It was assumed that in the case of polymerisation in monomer droplets the polymer will be coloured and when it takes place in solution it should be colourless. If polymerisation takes place in more than one site the product should be coloured inhomogeneously.

Sudan Red was chosen as the dye because it is insoluble in water and experiment showed that it is stable to the initiators used and has only a small inhibiting effect on the polymerisation process.

To determine the effect of the nature of the initiator vinyl chloride was polymerised in the presence of gelatin with (a) potassium persulphate (water soluble), (b) azobis-isobutyronitrile (partially water soluble) and (c) benzoyl peroxide (insoluble in water).

System (a) gave colourless polymer with a minor amount of deeply coloured particles. The bulk of the dye was expelled to the surface of the water. System (b) gave a mixture of deeply dyed and colourless particles (about 1:1) with a minor proportion of polymer of varying depth of colour. System (c) gave coloured

polymer with a very small number of colourless particles. Viscosity measurements indicated that the molecular weight of the coloured polymer was identical with that of polymer obtained in control, bulk polymerisations, whereas the colourless polymer was higher in mol. weight.

Other monomers differed in the ratio of coloured to colourless polymer formed but the picture was similar.

Experiments on reaction rates in the absence of monomer droplets, studied by adding vapour phase monomer to the colloid solution, showed that the rate under these conditions was the same as in ordinary suspension polymerisation. This suggests that polymer formation is the rate determining factor in suspension polymerisation, re-establishment of monomer concentration in solution being more rapid.

From their results the authors reject the idea of microbulk polymerisation as a satisfactory explanation of suspension polymerisation.

HOUDRY PROCESS

NEW routes to propylene and isoprene are now likely with the Houdry dehydrogenation process. The process was first introduced during the last war for the production of butadiene from n-butane. It converts light paraffin hydrocarbons to corresponding mono- and di-olefins with minimum side reactions. The preheated feedstock is fed to refractory-lined carbon steel reactors. The endothermic reaction takes place in a chromia-alumina catalytic bed at about $1,000^{\circ}$ F.

Effluent from the hot reactor is oil quenched, the light gases are separated and enter the plant fuel system. In the olefin extraction section, the desired fractions are separated and the raffinate is recycled to the reactor feed.

High molar selectivity for propylene from a predominantly propane stock is claimed from pilot-plant investigations. A 77 per cent propylene product stream is obtained from a 96 per cent propane feed stream. A typical catalytically cracked C₅ fraction is found to consist of isonand n-pentanes and pentenes which yield 41 per cent isoprene.

Polyprene is, of course, of marked interest at present, for direct polymerisation. Interest in isoprene has been aroused by recent news of 'natural-type' synthetic rubbers.

New Chemicals

CELLULOSE ACETO-BUTYRATE FOR WHIRL SINTER PROCESS

CELLULOSE aceto-butyrate material is now being offered by R. H. Cole and Co. Ltd., 2 Caxton Street, London SW1, in powder form suitable for the whirl sintering process of coating metals. This material, the trade name of which is 'Cellit sintering powder', is initially intended for supply in unpigmented form only; recommendations will be given on request for suitable colouring formulations.

Cellit sinter powder is said to be suitable for the production of high gloss coating on all types of metal objects made from steel, cast steel, cast iron, light metals, copper, etc. In addition to imparting good chemical resistance, these coatings have good electrical insulation properties and are claimed to be very resistant to impact.

The plastics coating is applied by whirl sintering, for smaller objects a special flame spraying technique has to be used. Coating is carried out in a Griesheim spray sintering apparatus in which the powder is whirled by blowing either air or nitrogen through a porous base plate. Blown air should be free from water, oil and dust. The amount

needed for a powder depth of less than 800 mm., is 0.35 to 0.45 m³/h. and dm² surface area

Pre-heating temperature of the metallic parts depends on the form, thickness and nature of the material to be coated. It will normally lie within 320-360°C, Coating thickness depends on pre-heat temperature and length of dipping time. Cooling of the articles in air gives a high gloss surface.

For colouring R. H. Cole and Co. recommend that the dry dyestuff be stirred with a part of the Cellit sinter powder. Mixing is then completed in a fast running mixer, a mixing time of 10 minutes in a paddle mixer giving a completely homogeneous powder.

The following technical data has been issued on soft grade Cellit sinter powder: specific heat Kcal, 0.35; loose weight g/1, about 350; practical cold resistance, -40°C; practical heat resistance about 100°C; melting range about 180-190°C; range of particle size, 0-150 microns; heat conductivity, kcal/mh°C 10², 17; breakdown resistance, kV/mm, 20.

First UK Plant To Produce Expanded Polystyrene By Continuous Moulding

EXPANDED polystyrene is produced by a continuous moulding process for the first time outside the US, at a new plant commissioned recently at the works of Styrene Products Ltd., Partington, near Manchester. With a daily capacity of more than 4,000 cu. ft., the plant is operated under licence from W.M.B. International, AB, Sweden, from whom Styrene Products (an associate of Shell Chemical) have the exclusive rights in the UK and Commonwealth, excluding Canada.

Registered as Styrocell, the new product is made from polystyrene beads incorporating an expanding agent which, on the application of heat, expands the beads to more than 60 times their original size. The beads are then fused to give a finished product in the form of light-weight rigid boards or blocks of multi-cellular structure. The cells are non-communicating, contain air and the boards have exceptional thermal insulating properties. They resist the passage of water vapour, are white and show good mechanical strength.

Expanded polystyrene is widely used as an insulating material in cold storage rooms, deep freeze rooms, cold lockers, road and rail containers, etc. It also has a large potential outlet in the building trade as an insulating material; as a sound transmission barrier; and as anti-

condensation membranes in walls, floors and ceilings. It is also used in sandwich construction as a core between facings of many different types of constructional material.

'Styrocell' is available in standard and self-extinguishing grades, the standard grade having a density of 1 lb. per cu. ft. nominal.

ICI Introduce Two New Dyestuffs

Two new dyestuffs from Imperial Chemical Industries Ltd. are Procion Scarlet G, the latest addition to the expanding range of Procion specialities, and Carbolan Rubine 2B, a new homogeneous acid milling dyestuff.

Main features of Procion Scarlet G are its ability to produce full, bright, yellow-ish-scarlet shades of good fastness to severe washing on cellulose fibres, good fastness to light on viscose rayon and good batchwise dyeing properties on cellulosic fibres generally.

Carbonal Rubine 2B is of interest for the economic production of heavy shades on loose wool and slubbing. Exhaustion obtained under the weakly acid conditions usual in these applications is said to be very good. The dye is suitable for the high-temperature (pressure) dyeing of wool

Price's Produce New and 'Unequalled' Oleine with Wide Use Range

PRIOLENE 95, one of a new series of oleines introduced by Price's (Bromborough) Ltd., Bromborough Pool, New Ferry, nr. Birkenhead, is stated to be unequalled in its type in any country in the world. Described as a new concentrated oleic acid with a remarkably low content of polyunsaturated and saturated fatty acids, it is suggested for use in disinfectants, duplicator stencils, esters, emulsifiers, rayon finishes, shampoos and textile lubricants.

Tentative specification of Priolene 95 is: titre 13.5-15.5°C; iodine value 87-90; acid value 198-201; saponification value 198-201; 'unsaponifiables 1.5 per cent max.; colour (Lovibond units 1 in. cell) 2.0 Y, 0.4 R max. Typical composition is: monounsaturated acids 92 per cent; polyunsaturated acids 2 per cent (linoleic acid 1.5 per cent, linolenic acid 0.5 per cent); saturated acids 5 per cent; unsaponifiables 1 per cent.

All the principal grades of the new Priolene range of oleines are available to three different colour standards to enable buyers to make separate decisions on the specification and colour standard.

This new range is parallel to the Pristerene range of stearine announced two months ago. Price's new integrated production processes now include the only two Emersol plants for the solvent separation of fatty acids in the UK.

New Pyridine Compounds From Midland Tar

RECENT additions to bulk chemicals produced by the Midland Tar Distillers Ltd., Oldbury, nr Birmingham, are: 4-t-butylphenol; 3-picoline; 4-picoline; and 2-6-lutidine. A new booklet, entitled 'New Organic Intermediates', lists a number of chemicals, largely alkylphenols and pyridine derivatives, now produced in the company's special products department. Including many chemicals not previously available in the UK, these are available in development quantities varying from lots of 10 lb. to 5 cwt.

Suggested commercial use for alkylphenols are in the preparation of plasticisers, antioxidants, selective weedkillers, lubricating oil additives, wetting agents and detergents, and phenolic resins, etc.

In the section dealing with pyridine derivatives, it is stated that pyridine N-oxides and the methylpyridine N-oxides are the first to be made commercially available in this country. It is thought that their novel chemical properties will offer new prospects in pyridine chemistry. 2-Vinylpyridine can be readily polymerised and copolymerised with a variety of monomers to give products of value in fibres, photographic films, adhesives and ion exchange resins. A number of compounds will add across the double bond of vinylpyridine to give pyridyl-ethyl derivatives.

Washington Chemical to Extend

Considerable factory extensions, to provide for the manufacture of insulating materials, are proposed by the Washington Chemical Co. Ltd.



materials, in addition to other products, will be developed from them.

The research team was headed by Mr. N. L. Paddock. A detailed booklet on the phosphonitrilic chloride polymers is available from the company's organic chemicals department at 1 Knightsbridge Green, London SW1.

THE USE of rocket fuel as an antidepressant for laboratory animals would seem to come into the shock-treatment category. The news that this project was now undergoing extensive clinical trials for the treatment of the depressed state in human beings made me reach for the proverbial pinch of salt.

But I have it on the authority of Dr. B. Biel, a senior chemist with Lakeside Laboratories Inc., Milwaukee, US. that chemicals derived from the rocket hydrazine have shown powerful stimulating action against depression and fatigue in laboratory animals. The most promising member of the group, 1-phenyl-2-propyl hydrazine (code name JB-516) is the one now being tested on men and women. It acts by directly stimulating the central nervous system and by blocking the brain enzyme that destroys adrenalin and other activating hormones. The chemical JB-516 has been shown in laboratory tests to inhibit the brain enzyme for an entire week. There is, says Dr. Biel, no following period of dep ssion, a tendency to lower blood pres ure and to increase the appetitie.

Several hundred hydrazine derivatives of the sympathomimetic amines have been synthesised. The compound JB-516 is said to differ from iproniazid, the first potent monoamine oxidase inhibitor discovered by being a more potent inhibitor by 20 to 30 times.

FULL CREDIT must be given to the team of research chemists working in Albright and Wilson's central research laboratories on a project into the nature and properties of phosphonitrilic chlorides, who have made possible the commercial separation of the trimer and the tetramer from the crude mixture of cyclic and linear polymers. Sample quantities of pure trimer and tetramer are already available and greater quantities will soon be available. This is a notable advance in polymer chemistry for the company are the first in the world to announce the separation of the polymers on a commercial scale.

In recent years, the phosphonitrilic series of inorganic polymers has attracted increasing attention from research chemists. This has been largely due to the demand for high temperature-resisting materials, typified by the recent spurt in both rocket and space research where materials are required to maintain their properties in spite of exposure to extremely high temperatures,

The phosohonitrilic compounds have several similiarities to silicones, manufacture of which was pioneered by the company in the UK, and A and W hope that a wide range of heat-resistant

THE USE of inactive tracers, of wide potential application in production processes where radioactivity in the product has to be avoided, was the subject of a number of papers discussed in a recent symposium arranged by the Society of Glass Technology and the UK AEA Atomic Energy Research Establishment, Harwell (Journal, Soc. Glass Technology, 55-118, 205, 1958).

The quantity of selenium left in a

The quantity of selenium left in a glass after the addition of a decoloriser can be determined by irradiating in a nuclear reactor samples of the glass with and without the Se additive. By using a differential type of scintillation spectrometer, Se contents of 10 p.p.m. are readily determined.

Using tantalum as a non-radioactive tracer, both the rate of throughput and the degree of mixing in a glass melting tank furnace have been assessed. After the addition of a small quantity of tantalum pentoxide to the glass batch the relative amounts of tantalum appearing in the product can be determined by irradiating samples in a high flux of neutrons, followed by measuring the activity induced in the tantalum present in the glass.

Sand labelled with an equilibrium mixture ¹⁶⁰Ba and ¹⁶⁰La have been used to trace the passage of sand through a sand washing and grading plant and thence through a glass-melting tank furnace.

REPORTS suggest that all is far from well with Venezuela's ambitious £100 million State petrochemicals complex comprising a petroleum refinery, caustic soda and chlorine units (German built), and facilities for the production of synthetic fertilisers (Italian designed), insecticides, explosives, natural gas, synthetic rubber, plastics and synthetic fibres. Site work started in 1955 and construction began in the summer of 1956. First units came on stream towards the end of 1957.

The Petrochemicals Institute of Venezuela now states that this project has so far cost Bs446 million (£47 million), of which Bs98 million (£9 million) is still unpaid. Not only is it officially considered that the expenditure so far has been to date much higher than was neces-

sary, but that some completed sections of the plant will not function properly.

Despite all this, the Government is to go ahead with the project now that it is so far advanced. Doubtless the Venezuelan authorities would have been a little more careful in their planning, estimating and costing if they had been answerable to a group of shareholders.

NEXT STAGE in the £5 million rebuilding programme of Joseph Crosfield and Sons Ltd., Warrington, will be the demolition of the wharf warehouse and the rebuilding of the research and technical services block. The whole project is expected to be completed by 1965.

Last week we reported the first stage, when Mr. Ralph E. Huffam, a director of Unilever and a former director Crosfield, laid the foundation stone of the new chemicals building. Now I learn from Dr. J. E. Taylor, chairman of Crosfield, that although the application of production study technique and instru-mentation would minimise labour effort in the new building, it is not expected that this will lead to any redundancies in the 3,000-strong labour force. In the investment of £5 million, Crosfield and Sons foresee an expansion in trade that will take up any labour that might be saved with the introduction of automatic process control.

The ultimate aim is to concentrate production on the Lancashire side of the Mersey to avoid the cross-river movement of personnel and materials.

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THE increasing use of barrier creams in industry has led to a number of experiments being carried out in an attempt to improve their efficiency. British Oxygen Co. now claim in Patent 797,992 that the emulsion type of barrier cream can be improved by the addition of small quantities of p.v.p. (polyvinyl-pyrrolidone). It is said that the p.v.p. has the effect of enhancing the protective qualities of the cream. In addition, it gives a softening action which persists even after the hands have been washed.

The addition of p.v.p. substantially assists the removal of dirt or grease and the hands are left in a particularly clean condition. Experiments carried out in the laboratories of British Oxygen Research and Development Ltd. at Morden have shown that the correct amount of p.v.p. to use is between 0.5 and 1.5 per cent of the total weight of the cream. The invention relates only to the 'emulsion type' of barrier cream which is in the form of an aqueous or watery emulsion, the nonaqueous phase of which consists of greasy, fatty or waxy substances.

I learn that British Oxygen are market-

I learn that British Oxygen are marketing pilot quantities of the raw material and would welcome enquiries from barrier cream manufacturers who might consider incorporating p.v.p. in their product.

Alembic

GERMANY'S FIRST 'COLD' RUBBER PLANT



The Bunawerke plant at Marl from the air. Work began on the installation, which adjoins the works of Chemische Werke Hüls, in the spring of 1956

Bunawerke Hüls Unit has Capacity for 45,000 Tons a Year

EST GERMANY'S four major chemical producers, Chemische Werke Hüls AG, Badische Anilin- und Sodafabrik AG, Farbenfabriken Bayer AG and Farbwerke Hoechst AG, on Monday saw the coming into operation of a joint plan. On that day, Germany's first plant for the manufacture of 'cold' synthetic rubber was opened at Marl, Westphalia, by a company which they set up three years ago as a joint venture, Bunawerke Hüls GmbH. Chemische Werke Hüls, on whose ground the plant has been built and who will work in close co-operation with the operating company, holds a 50 per cent interest in it. The other three concerns, all successors of the liquidated I.C. Farben company, hold 16\frac{3}{2} per cent each.

Bunawerke Hüls, for whom a capital expenditure of DM 180 million (about £ $1\frac{1}{2}$ million) has been made by the mother companies, who have also transferred some of their top experts to the new company, plans to produce 38,000 tons of butadiene annually. This will be copolymerised with styrene made on the adjacent premises of Chemische Werke Hüls. Some 45,000 tons of synthetic rubber bearing the trade name of Buna will be produced. This capacity figure can be raised to 70,000 tons a year.

The plant has been working itself in slowly since June of this year, and at present butadiene and rubber are being turned out at only about 40 per cent capacity. When the plant has got into full swing it is estimated that it will be producing amounts of Buna equivalent to a quarter of the West German demand for all kinds of rubber. At present nearly all rubber has to be imported from the US. The tyre industry will take some 55 per cent of the Marl plant's output.

Work at Bunawerke is divided into two main stages—production of butadiene and its combining with styrene by an emulsion polymerisation process for its final preparation.

For the production of butadiene, which is present at about 70 per cent in the synthetic rubber product, Hüls decided on grounds of cost to work to a dehydration process developed by the American Houdry Process Corporation rather than use a method using the same base, *n*-butane, patented by the Phillips Petroleum Co. By the Houdry method, the working of the *n*-butane (CH₃-CH₂-CH₂-CH₃-> CH₃-CH-CH=CH₂+2H₂-67.0 cal), is undertaken in a reservoir-heated catalysing chamber at a temperature of about 600°C and a pressure of 0.15 ats. The catalyst used is a chrome-activated aluminium oxide. Vacuum pumps are used to reduce pressure of the hydrocarbons.

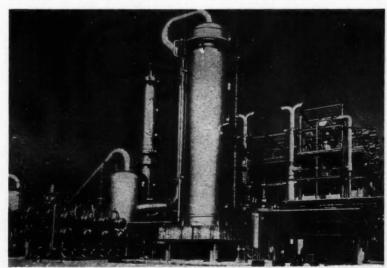
The butane yield with this method is said

to be about 55 per cent. This is a poorer yield by about 10 per cent than that obtained from the Phillips process, but even taking this into account, Bunawerke considered that the far higher initial and running costs of the Phillips process would

make it less practical than the Houdry method. In both methods catalysing has to take place due to the presence of C₄ hydrocarbons, and as this can be dehydrated in part only, provision has to be made for the withdrawal of liquid gas remaining after the process for re-treatment.

Another primary problem which faced the company was whether to use *n*-butane for the production of butadiene, or *n*-butylene. It had to be borne in mind that, with the steady growth of steam-cracking in Germany and elsewhere, more and more *n*-butylene would be coming into existence and could be worked into butadiene. However, *n*-butane was chosen both on grounds of efficiency as well as the fact that it is at present produced in crude oil distillation and hydroforming processes in sufficient quantity to supply any foreseeable demand in West Germany for use in any form of synthetic rubber manufacture.

To ensure a steady flow in the process, dehydration is carried out in two sets of three reactors, representing two streams of butadiene; at present, only one stream is being worked. After the catalysing process has taken place in these reactors, the resultant product is passed through a cooling tower, an absorbing tower, where waste



The dehydration plant for the production of butadiene. In the foreground is the cooling tower and behind it and to the left is the propane tower where the butadiene receives a primary cleansing and is separated from C₃

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gas is drawn off, and a matching desorber. C4 is then separated from C3 in a propane, or purifying column, and the C4 goes on to a row of 10 purifying units, each 13ft by 43ft and containing a solution of copper ammonium acetate. Using the CAA process of the US Esso company, Bunawerke here first wash the butadiene free from the mixture in which it has travelled from the catalysing unit and then, in settling tanks, separate it from the butylene with which it is mixed. After the butadiene has been brought to the required concentration it is piped to storage tanks ready for polymerisation.

The building-up of the synthetic rubber molecules themselves is achieved by copolymerisation at the low temperature of This emulsion polymerisation is a development of the defunct I.G. Farben concern, though synthetic rubber previously made by Chemische Werke Hüls was of the more usual 'warm' type. During polymerisation ammonium vaporising pipes keep temperature down to the required level. So sensitive is the polymerisation plant to impurity that only 60 per cent of the butadiene-styrene is treated; the remaining 40 per cent is passed back to a cleansing and rectifying plant, then being fed back to the polymerisation plant at a higher quality.

The final process is to make the latex produced by polymerisation into a solid rubber form. The latex fluid is, after an initial stabilising, precipitated with electrolytes. The Buna passes through three stirring vats in the precipitation process, in the last of which it is washed neutral and channelled to spiral presses for a primary draining process. Disc-grinders disintegrate any sludge and the Buna-crumbs are carried by conveyor belt along a warm-air channel where they receive their final drying. The crumbs are dropped into a press, which is set to compress them into block form every time 25kg (about 55lb) have fallen into it. This occurs about every 90 seconds and the block of synthetic rubber is then directed along belts where it is automatically wrapped in a plastics cover and dropped into a paper sack. It is then delivered by automatic truck to the dispatch depart-

There is a high degree of mechanisation and the labour force totals only 300.

Polidrum Cleared for Home and Export Transit of Some Corrosives

WO series of tests carried out on the Two series of tests carried out on Polidrum, a polythene-lined drum, in all three sizes: 5, 10 and 12 gall, have been given a clearance for both home and export transit of certain dangerous liquids. London Containers and Noakes Ltd., Beta Works, Butchers Road, London E16. point out that the authorities concerned do not give a 'blanket' clearance and any substance not already approved must be submitted for their consideration. The company adds that in this respect a number of corrosive fluids such as hydro-fluoric acid have already been approved.

The tests carried out by the Package Testing Station of the Printing, Packaging and Allied Trades Research Association at Leatherhead were suggested by the Railway Clearing House to evaluate

the performance of the Polidrum during simulated 600 mile journeys. space. Tests included continuous vibration on a jolting table; intermittent vibration; inclined plane tests (representing deceleration on impact of a truck striking buffers at 4 m.p.h.) and drop tests on to a steel plate from a height of 2 ft.

At the end of the trials Patra stated that no damage appeared to have occurred to the inner polythene container. The screw-cap remained firm throughout and no leakage had taken place.

Following these tests the Railway Clearing House have notified their approval of the Polidrum for general traffic. The other tests, carried out at the request of the Ministry of Trans-

were filled, allowing 10 per cent ullage

Inset are two views of Polidrums after drop testing on to a section of railway line. These tests, said to be of 'a severity to which no package has previously been subjected', were made from the special gantry. A cordoperated release gear was used. With other trials by Patra, they were designed to obtain export approval from the Ministry of Transport and home trade approval from British Railways for the carriage of dangerous fluids

port, involved dropping the drums, vertically and horizontally, on to a section of railway line from heights of 10, 15 and 20 ft. The tests were independently observed by and reported on by the representative of a chemical organisation.

The intention was to simulate conditions of hazard that might obtain if a crane or dock pallet failed while a ship was being loaded. The Polidrums were of 22 B.G. outers and contained water plus 10 per cent ullage space.

As a result of this series of tests the largest size of Polidrum was found to resist satisfactorily drops from 10 ft. A moderate proportion of 'leakers' likely when dropped from 15 or 20 ft., but where leakage occurs, the area of splash-spread is not likely to exceed a radius, from the centre of impact, of the order of 3 ft. Throughout the trials all screw-caps are reported to have remained firm and leakproof.

The Ministry is now willing to consider applications by chemical manufacturers for the approval of the Polidrum for the shipment overseas of 'dangerous corrosives'. Up to the present, approval has been given for a number of dangerous liquids including: hydrofluoric acid, hydrochloric acid and formic acid.

It is stated that these particularly severe trials were intended to establish safety factors and to evaluate the strength of the Polidrum. They were also in tended to assist consideration in any reframing of current regulations deemed necessary by the Ministry should it decide that carriage of dangerous corrosives in Polidrums might merit a more convenient stowage position when ship-

Geigy Produce Four Phthalocyanine Blues

GEIGY have completed the first stage in their programme of manufacturing phthalocyanine blue in the UK. The modern plant built for this purpose at the Paisley works of James Anderson and Co. (Colours) Ltd. has been in operation since last year and has shown that the phthalocyanine colours can be pro-

duced satisfactorily and consistently.

Four different standards of copper phthalocyanine blue are now available. Irgalite fast brilliant blue BNL is a strong red-shade blue for printing inks and unplasticised plastics such as polystyrene. Of excellent texture, it disperses easily and completely, and is available in paste form. It is not stable in the presence of aromatic solvents.

BGL is the stabilised version of BNL, and a general purpose red-shade blue for paints. It has excellent texture and the highest fastness properties. A newer type of copper phthalocyanine blue, GLS, gives a greener shade of blue which makes it possible to produce very clean shades in paints and inks. It is said to be completely solvent-stable, and shows a reduced tendency to flocculate.

Irgaplast brilliant blue BLP was developed for plasticised plastics such as polyvinyl chloride and cellulose acetate, in which it disperses readily. It has excellent heat and migration stability.

Birmingham Microchemistry Symposium-4

CHEL 242 as a New Complexing Agent in Titration and Masking

AN organic complexing agent closely related to ethylenediaminetetraacetic acid (EDTA) was CHEL 242. It had, however, a remarkable characteristic reaction in that it formed a colourless strong complex with iron in strongly alkaline solution. Discussing this reagent, which is produced by Geigy Industrial Chemicals Inc., Ardsley, New York, US, and whose formula is still undisclosed, Dr. K. L. Cheng (Metals Division, Kelsey-Hayes Co., New Hartford, New York, US) described it as a non-hydroscopic white powder with a pH of 5 in 1 per cent aqueous solution. The sodium salt (CHEL 242 PN) was very soluble in water and acid but was only slightly soluble in concentrated HCl and ethanol.

From its reactions with metals, it was believed that this agent was also a polyaminecarboxylic acid. In slightly acid, neutral, and slightly alkaline media it behaved similarly to EDTA, except that the stabilities of its metal complexes were slightly weaker. It could not take the place of EDTA in all complexometric titrations or in masking, but it did offer certain advantages over EDTA.

Titrations of metals with CHEL 242: CHEL 242 formed weaker complexes with the alkaline earths than with copper and zinc but formed a quite strong complex with aluminium. Titration of aluminium and zirconium could be made at room temperature.

Back Titrant Unsuccessful

Although CHEL 242 formed a complex with titanium, an attempt to back titrate the excess CHEL 242 with ferric chloride using salicyclic acid as indicator (Milver and Woodhead's method, Analyst, 1954, 79, 363) was not successful since ferric iron displaced titanium rapidly at pH 4.0 to 6.5. Because of this aluminium could be titrated in the presence of titanium by using ferric chloride to back titrate the excess CHEL 242 at pH 4.0 to 6.5 using salicyclic acid as indicator. To titrate titanium or the total amount of titanium and aluminium, copper sulphate was used as the back titrant and PAN as indicator with pH at 4.0 to 4.8.

Reagent: CHEL 242. Approx, 0.05 M solution standardised with a standard iron (III) solution using salicylic acid as indicator at pH 4 to 6 or standard copper solution with PAN as indicator at pH 4 to 5. Standard iron solution: 0.05 M solution prepared from pure iron or from an iron salt, standardised with standard EDTA solution. Standard copper solution: 0.05 M solution prepared from pure copper or from a copper salt, also standardised with a

This report concludes our coverage of the International Microchemistry Symposium at Birmingham. Other reports appeared in 'Chemical Age' 30 August and 6 and 13 September. Full proceedings will be published by Pergamon Press

standard EDTA solution. PAN: 0.2 per cent in methanol; salicylic acid: crytal.

Procedure for aluminium (A): To a solution containing 0.1 to 0.5 millimole aluminium, a slight excess of CHEL 242 was added. After addition of 3 gm. of ammonium acetate, the solution was adjusted to pH 4 to 5.5. About 0.2 gm. salicylic acid was added. Excess CHEL 242 was back titrated with a standard ferric chloride solution. End point was from yellow to reddish brown.

Procedure for total aluminium and titanium (B): Slight excess of CHEL 242 was added to a solution containing both metals. After addition of 3 gm. ammonium acetate, the solution was adjusted to pH 4. Five drops of 0.2 per cent PAN solution were added. The solution was back titrated with standard copper solution. The end point was when the indicator changed from yellow to brownish red and stayed for at least 10 to 15 seconds. The amount of titanium was obtained by the differences between the two titrations (B—A).

Cu and Zn: Similar to EDTA procedure; direct titration using PAN as indicator.

Fe: Similar to EDTA procedure; direct titration using either salicylic acid or Tiron as indicator.

Zr: Similar to Körll and Pribil's EDTA procedure. Titrated at pH 1 using Xylenol Orange as indicator. No heating required.

Ca and Mg: As good as EDTA in calcium using Erichrome Blue Black R. C1202 (Eriok) as indicator. For magnesium, Erichrome Black 7 (Erio T) was used as indicator.

Rare earths: By addition of slight excess of CHEL 242, and back titration using copper sulphate and PAN as indicator at pH 4 to 10.

Precipitation of metals with NaOH in presence of EDTA and of CHEL 242: Four drops of 0.01 metal solution were mixed with four drops of 0.02 M EDTA or CHEL 242 solution (pH 5 to 6). One or two drops 10 N NaOH were added. Appearance of turbidity was considered as a positive test.

Be, Mg, Ag and U (VI) formed precipitates with NaOH in presence of either EDTA or CHEL 242. Ti, Zr and Hf

formed white precipitates in presence of EDTA but Zr and Hf in strong alkali in presence of CHEL 242 at room temperature formed no precipitate. Iron (III) formed a reddish precipitate in presence of EDTA. The yellowish coloured ferric solution changed to clear colourless solution upon addition of NaOH in presence of CHEL 242. Ferrous iron formed a similar solution in presence of NoOH and CHEL 242.

Silver was precipitated by NaOH in presence of either EDTA or CHEL 242. Addition of CHEL 242 unpurified solution to either AgNO₃ solution or to mercury (II) nitrate solution caused heavy precipitates (pH under 5) which persisted upon addition of NaOH. Like EDTA, CHEL 242 prevented the NH₃ test by Nessler reagent. Purified CHEL 242 precipitated neither silver or mercury (II). Sn, Sb, Cr (III) solutions in presence of either EDTA or CHEL 242 and NaOH remained clear.

Addition of CHEL 242 to tellurium (IV) solution (pH approx. 5) caused a white precipitate, soluble in Na₄OH or NaOH. The precipitate also dissolved on heating but on cooling turbidity of the solution was observed.

Complexes with Cerium

CHEL 242 formed a very strong complex in the cerium (III), but not with cerium (IV); these were precipitated with NaOH. Other rare earth metals were also precipitated under similar conditions; however, in an excess (200 to 300 per cent) of either complexing agent, the solutions were clear CHEL 242 complexes might be slightly weaker than those of EDTA complexes, it was thought.

Magnesium formed magnesium hydroxide with NaOH in presence of either EDTA or CHEL 242. The thiazole yellow test could be made in presence of either. Gold (III) was reduced to 1ts metallic form by either agent upon addition of NaOH: CHEL 242 was the more sensitive in this test.

Reaction of metals with H_2O_2 in presence of EDTA and CHEL 242. Ti, V, Cr, Mo and U gave coloured reactions with H_2O_2 in presence of either complexing agent. Coloured complexes were also formed with Fe (III)—CHEL 242 and CO (II)—CHEL 242 in ammoniacal media, iron (III—CHEL 242 coloured complexes resulting from addition of H_2O_2 in carbonate medium was unstable. For the ferric-CHEL 242 complex, perborate functioned similarly compared with H_2O_2 . These colour reactions were stated to strongly indicate that CHEL 242 was a polyamine-carboxylic acid.

Reaction of ortho, ortho-dihydroxyazo dye with metals in presence of EDTA and of CHEL 242: Four drops of 0.01M metal solution were mixed with eight drops of 0.01 M EDTA or CHEL 242. Solution was adjusted to pH 10 with NH₃OH and NH₄Cl buffer (pH 10) and one drop of 1 per cent Eriochrome Back T in methanol was added. The mixture was boiled. Colour of the solution was observed before and after boiling.

bonning.		
Metal	Colour	Remarks
Vanadium (V)	Deep red	Coiling.
Titanium (IV) Zirconium	Red	**
Hafnium Palladium	99	**
Platinum (IV)	Yellowish- Green	Bleaching the dye.
Gold (III)	Grey-black	Reducing gold on heating.
Tin (IV)	Red blue	Red colour appeared for short time, then changed to blue.
Tin (II)	No colour change	Tin was hydrolysed.
Uranium (VI) Thorium (IV)	Slightly red Deep red	Room temperature.
Gadolinium Cerium (IV) Iron (III)	Slightly red Yellow Yellowish- Brown	Due to impurity of Th. Bleaching the dye. Boiling.

Cheng suggested that the colour reaction of thorium might find application for qualitative and quantitative determination of thorium, zirconium, etc., in the presence of many other metals. Erio R was not as sensitive as Erio T for thorium.

CHEL 242 as masking agent: Like

EDTA CHEL 242 was found to act as a masking agent in reactions of metals with diethyldithiocarbonate. Ag, Hg, Th, Cu, Bi, Pb, Pd, Pt, Os, and Cd formed white or yellow to brown precipitates in presence of either agent at pH 6.5 and pH 10 to 11 respectively. Excess of either agent prevented precipitation of Cd diethyldithiocarbonate at pH 10. Tellurium did not form any colour cr precipitate with diethyldithiocarbonate at pH 10 but did at lower pH values. Iron (III) formed a red complex with Tiron in presence of both agents in ammoniacal media (pH 10 to 11). No iron Tiron complex formed in weakly acid media. Titanium (IV) reacted instantly with ammoniacal solution of CHEL 242. Rapid formation of titanium Tiron complex occurred in presence of either masking agent in weakly acid

In general CHEL 242 was found to react faster than EDTA with certain metals (e.g., Al and Zr). In alkaline media, it had definite advantages in not hydrolysing Fe, Ti, Zr, and Hf. In certain cases it would be an advantage to use CHEL 242 as masking agent.

Carbon-Hydrogen Determination Paper Provokes Discussion

THE object of the short lecture by Dr. W. Schoniger (Sandoz Ag, Basle) on 'Some aspects of carbon-hydrogen determination,' was to provide different productions of the state o

mination, was to provoke discussion.

Dr. Schoniger said that in the carbon and hydrogen determinations, two values were obtained from one analysis which were significant for most compounds. Pregl had developed his method and this had been used for 50 years. However, during the last few decades, many new methods and techniques had been developed. These could be divided into three types.

(1) Modifications in the conditions for combustion.

(2) Modifications in the way in which nitrogen oxides are removed.

(3) New methods.

Modifications in the conditions for combustion: In fitting the Pregl apparatus, exact conditions must be used for accurate results, especially for 'hard burning' substances. In the last 20 years, specific catalysts had been developed. In high temperature furnaces, temperatures of 800°C + were employed for the empty tube method and the substance was burnt in a fast stream of oxygen. Russian workers were using methods based on this, it was reported. Kirsten had also tried this with such a method and high temperatures automatic combustion was possible.

In the last two years, two methods had been developed which used rather low temperatures. In one the catalyst employed was new—silver pervanadate—and thermal decomposition. In the other cobalto cobaltic chloride and asbestos was used. Both methods were described as very good. Tube fillings which permitted the determination of other elements were Si, P, F, N, etc.

Modifications in the way in which nitrogen oxides were removed: Lead dioxide was the most important in the classical method. Other methods of absorption were:

(a) The use of sulphuric acid, diphenylamine, manganese oxide.

(b) Removal by reduction.

(c) In the US, ammonium sulphamide had been used and other workers had employed trihydroxy ammonium phosphate between the water and carbon dioxide tubes for the absorption of nitrogen.

New Methods: Carbon dioxide was still mostly determined gravimetrically. Water was determined by Karl Fischer. Carbon dioxide was absorbed in NaOH and precipitated as barium carbonate, dissolved in hydrochloric acid and barium iodate and titrated.

The most successful method in determination of oxygen appeared to be that of Unterzaucher. Combustion was carried out in air and the carbon monoxide was frozen out in water. When the water was warmed, the carbon monoxide came off.

Recently combustion and gas volumetric or manometric measurement could be used for labelled compounds — C¹⁴ being determined by radioactivity. Fractionation of the products by gas liquid chromatography was a possibility. Dr. Schoniger suggested that it would be useful to have an internationally accepted set of standards for testing methods, etc.

Dr. W. J. Kirsten (Uppsala Universitets, Uppsala, Sweden) informed Dr. Schoniger that he had tried a number of different combustion methods and found that at first good results might be obtained. However, after publishing a

note on one's method, other workers sometimes found that the method did not work and even the originator found that he could not analyse their samples either.

He had returned to the empty tube method and low temperatures of combustion in a difficult-to-complete combustion of long chain saturated compounds. Catalytic reactions had a tendency to be specific and might not always work.

At high temperatures, metallic halides might interfere with final determination. At 1,000°C he had experienced difficulty with the empty tube. He had thought that this might be due to silica. He had minimised this by keeping the furnace on overnight and had a reduced flow of oxygen when not in use.

Mr. G. Ingram (Courtaulds Ltd., Maidenhead, Berks) said he had been interested in catalysts in 1940-45, and particularly in the thermal decomposition product of Ag vanadate (one got an active mixture of silver and vanadium oxide). If a sample contained halogen or sulphur, the absorbent was retained. It had a disadvantage—e.g. fusion points—if used at 600°C and at higher temperatures it stuck to the tube. Later he had decided to use the rapid empty tube method. With a packed tube there was always the danger with routine work that the packing might get used up and low results might be obtained. Mr. Ingram said he was interested in the Russian technique.

Dr. Kirsten said he had experimented with the Russian method as an automatic method.

Mr. P. Gouverneur (Koninklijke-Shell Laboratorium, Amsterdam) agreed with the vanadate procedure. Its self-indicating properties were useful.

Fluorine Compounds

Dr. J. R. Hudson (Brewing Industry Research Foundation, Surrey) asked whether the empty tube method had been applied to fluorine compounds.

Mr. Ingram indicated that vanadate would retain fluorine compounds. He had obtained good results.

Mr. Blom (Statsmijnen in Limburg, Netherlands) said that in 1955 he had published a method in Analytica Chimica Acta in which CO₂ had been titrated in the C-H method. In 1952 he had examined the empty tube method and allowed 12 minutes for the determination. The method could be used for oxygen in organic substances and carbon in steel.

Mr. Ingram stated that a temperature of 900°C was not too high. The only trouble he had had was with a chlorinated diphenyl where he had had to raise the temperature to 1,000°C. But for this it had appeared that the sample did not decompose. If a piece of platinum asbestos was used instead of the quartz roll, the result was satisfactory.

Dr. Schoniger said he used silicone tubing on the apparatus to reduce breakages.

Mr. Ingram pointed out that in nitrogen oxide absorption, if a catalyst was used, it did not matter what the linkage Ltd dete Exe met omit the CO and obtathe rot met

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was. A high conversion to NO2 was obtained. With the empty tube method there was a much lower conversion of nitrogen oxides and there was an increased efficiency of absorbent. Advantages of the empty tube method are its simplicity, no maintenance is required and there is better retention of elements.

Dr. S. Gorbach (Farbwerke Hoechst AG, Frankfurt, Germany) said he was concerned with the rise in temperature caused by carbon and hydrogen apparatus. It was a question of insulation and this should be examined by the manufacturers.

Mr. Ingram informed Dr. Gorbach that he used a 11 in, thick insulator.

Dr. Schoniger wanted to know whether anyone had any experience with the Unterzaucher method. The general opinion on this method was that it was no good because of the time involved in carrying out a determination (75 minutes). It was satisfactory on the ultra-microscale.

There were two types of carbon and hydrogen determination. That carried out in the universities with relatively pure compounds and that carried out for industrial purposes where the sample might contain half the periodic table.

Dr. Gorbach considered that methods quoted in the literature should give the standard deviation obtained.

Mr. M. Attwood (Albright and Wilson (Mfg.) Ltd.) inquired whether anyone could compare the relative merits of the Flaschertager tubes and Pregl tubes.

Dr. W. T. Chambers (B.R.P.R.A., Welwyn Garden City, Herts) said that moisture absorption was about the same for both.

Mr. Ingram felt that Flaschertager tubes were best because he had found it necessary to wait for a longer period before weighing the tubes if the Pregl tube was used.

Physical Methods for Carbon Dioxide

Mr. C. Whaliey (Laporte Chemicals Ltd.) discussing the physical methods of determining carbon dioxide, said that at Exeter he had suggested that the gravimetric and titrimetric finishes should be omitted. He instanced the behaviour of the katherometer for determination of CO2 with no worry to date about water and oxides of nitrogen. A trace could be obtained which can be integrated to give the carbon dioxide. It was accurate but r.ot vet as accurate as the empty tube method (accuracy ~ 1 per cent). There were also the conductivity methods. He felt that there was considerable scope along these lines.

Dr. Gorbach said he had tried the Beckmann water absorber in carbon and

hydrogen determinations.

Mr. Johnson stated he had confirmed this electrolysis cell technique. He had used it to detect 1 p.p.m. in water in refrigerant gas.

Mr. Ingram thought it was obvious that analysts were on the brink of a change in carbon and hydrogen determinations.

Mr. Whalley suggested that instrument was yet to come.

ANALYTICAL POSSIBILITIES OF SCHONIGER FLASK COMBUSTION

DISCUSSING some analytical possibilities of the Schoniger flask combustion procedure, Dr. W. Schoniger said that analysts had to deal with organic chemists who would not believe an analysis and/or who require the analysis to be reported before the sample is received. With regard to the matter of choice of the right method, the accepted methods for determination of halogens and sulphur were too long. Most methods described in the literature used one of two ways for combustion-oxidation or reduction.

In the oxidation method, there was (a) burning in a stream of oxygen (platinum contacts): (b) the Carius method: and (c) the Parr bomb. In reduction, release of the halogen or sulphur was accomplished by hydrogenation, catalytically or by means of potassium or magnesium in sealed tubes. For the final determination of the halogen or sulphur, there were a number of methods available, but these were all time-consuming. They also had other disadvantages. The Parr bomb, for instance, on a micro-scale gave too much Na+ for a good potentiometric titration.

Three years ago, he (Schoniger) had contacted Micker and Pech who were using a litre bottle and burning about 0.2gm. of sample in an atmosphere of oxygen. He had queried whether this method could be used on a micro-scale. Micker and Pech had thought not, but he had tried it and was satisfied with it.

Absorption of Oxygen

Describing how the elements were determined after combustion, Dr. Schoniger said that normally, he waited 10 to 15 minutes for the absorption of oxygen by the products of combustion.

For chlorine, an argentiometric titration was used with dichlorofluorexein indicator in alcohol (Wagner) or mercuric hydroxy cyanate or a potentiometric method. For bromine, mercuric hydroxy cyanate was used but the factor was not good. It was better to oxidise to bromate and do an iodometric determination. For iodine, he oxidised to iodate and titrated with sodium thiosulphate.

To determine sulphur there were several methods: (a) if there was only CHOS in the compound, sulphur could be determined acidimetrically; (b) if CHOS halogen, the total acidity was determined and then the halogen by Hg(OH)CN; (c) if nitrogen was also present, the Flaschleas complex method could be used for titration of the sulphate; (d) sulphate could be determined conductometrically.

In fluorine compounds, some of the carbon-fluorine linkages would not break up. Phosphorus could be easily determined as phosphate as also C14 labelled compounds. It should be possible to determine arsenic and with regard to mercury, a paper was shortly to be published in the Analyst by a member of the present conference.

With regard to volatile compounds, Dr.

Schoniger thought that the device mentioned by Dr. Kirsten in his paper (polythene tubing) should be suitable. Of his flask method, Schoniger indicated that the flask should provide a very rapid method for qualitative analysis. He and his coworkers had carried out over 2,000 combustions and had so far had no explosion during the burning. The temperature of combustion was over 1,000°C and might be 1,200°C.

Miss M. Corner (National Chemical Laboratory, DSIR, Teddington) suggested that volatilisation before combustion might

occur by the lens technique.

Dr. A. Stev rmark (Hoffmann-La-Roche, US) had tried fluorine compound in the Schoniger flask, but had obtained low results. Using a mass spectrometer, CF4 had been found in the vapours. Last Spring, the products control group of his company had shown that with 50mg. samples, two or three times the amount of sodium peroxide was on the filter paper. On burning perfect results were obtained, even if there were three fluorine atoms on one carbon. The method was very satisfactory on a microscale. They were using 5mg. samples and 10mg. of peroxide.

Dr. R. Levy (Municipal Laboratory of Paris, Paris, France) thought that a potentiometric finish could be used to the Parr bomb

Incomplete Combustion?

Dr. W. Kirsten (Uppsala Universitets, Uppsala, Sweden) said he had obtained similar results with bromine and iodine and he thought it might be due to incomplete combustion. (He described experiments he had carried out to check the

Dr. Schoniger suggested that higher combustion temperatures or lower sample weights might help. A member of the conference had told him that a drop of decalin on the filter paper helped to give higher temperatures.

Dr. Kirsten stated that with large samples he had found that sometimes he obtained a thick soot and he had to decrease the sample size.

Dr. Schoniger indicated that minimum oxygen requirements' could be calculated from the weight of the sample and filter paper.

Mr. Cosgrove (Borax Consolidated Ltd., Chessington, Surrey) asked whether it would be possible to determine carbon by this method, using aluminium foil instead of filter paper?

Dr. Schoniger said he had tried this but was unsuccessful. If the sample was ignited from outside it might be possible, he

Dr. Colson (Alkali Division, Imperial Chemical Industries Ltd.) wanted to know whether Dr. Schoniger had used his (Schoniger's) method for boron?

Miss M. Corner answered Dr. Colson, saying that she used nothing else but that method for boron.

SOURCES OF ERROR IN MICROCHEMICAL WEIGHING

MICROCHEMICAL procedures have led to the development of highly refined instruments having a capacity approaching that of ordinary analytical balances, but a sensitivity about a hundred times better. The microchemical balance usually has a capacity of about 20 g. and is capable of detecting changes of mass of the order of 1 microgram. In his paper which was introductory to a discussion of microchemical weighing errors, Dr. G. F. Hodsman (L. Oertling Ltd., London) reviewed briefly some sources of error and suggested ways of mitigating their influence and assessing their effect on the reliability of a weighing operation.

Two main types of error were generally recognised as impairing the reliability of a result—random and systematic. The former led to a lack of precision in a result and the latter to a lack of accuracy. Magnitude of probability of error was assessed by computing the standard deviation, using the deviation of the individual results from

the mean.

Three groups of sources of weighing errors were considered: environmental; instrumental; and weighing technique.

Environmental Errors

Under environmental, Dr. Hodsman considered temperature fluctuations. With these, the beam might not expand uniformly, leading not only to rest-point changes but also to inequalities in the arm-length of the beam which might be irreversible. Undue fluctuations in atmospheric conditions also influenced reliability. The buoyant effect of dry air at 20°C and 760 mm. pressure was about 1.2 mg. per c.c. difference in volume between the loads in the two pans. A variation in temperature of 1°C or in pressure of 3 mm. changed this buoyancy effect by almost five micrograms. Variation in water vapour content of the atmosphere also influenced buoyancy.

Minimisation of these errors could be effected by housing the balance under reasonably constant conditions of temperature, pressure and humidity; tares should be used whenever possible to minimise buoyance influences, and weighings should be carried out expeditiously.

Excessive vibration was of importance since it led to deterioration of the knife-edge and to a loss of precision. If possible, vibration should be dealt with at its source. Lack of cleanliness both of a balance and its surroundings was

also mentioned.

Defects were likely to arise in an instrument after a period of use, which were not immediately apparent. Blunting of the knife-edge was considerably accelerated by injudicious operation of a balance. The position of the knife-edges in the beam relative to each other was significant. Loss of precision would also result unless the pans were always loaded exactly centrally.

Where the rider had been omitted from balances, errors due to loss of precision did not occur. To avoid weighing inaccuracies with microchemical balances a projected image of a graticule was fixed to the beam as a means of determining the smallest differences in weight. Weights were another source of loss of accuracy and intercalibration of the weights used might be necessary.

Differences in weighing techniques could influence the reliability of the

results. In particular, stress was laid on acclimatisation in atmospheric conditions similar to those obtaining in the balance case for vessels to be weighed. Static charges through wiping when the relative humidity of the atmosphere did not exceed about 50 per cent was important.

The most satisfactory assessment of weighing errors was to specify the standard error of the instrument. A check test should be repeated once a month and the results recorded. Any deterioration in performance of the balance then became evident. Where it was necessary to ensure absolute weight, three factors should be checked, namely: arm-length equality; sensitivity; and weights. For checking the last, the Richards method was most commonly used.

Micro-techniques and Apparatus Demonstrated at Birmingham

EMONSTRATIONS of micro techniques and apparatus were arranged at the International Symposium on Microchemistry with the co-operation of companies, research associations and universities, etc.

Various devices and apparatus were demonstrated by Dr. J. T. Stock, University of Connecticut, US, and Mr. M. A. Fill, Norwood Technical College. A transistor-operated dispensing device for liquids was shown. The device is used in conjunction with an electromagnetic control valve for dispensing fixed volumes of liquids such as indicators or stains, the filling of ampoules, etc., and it can be used for routine or remotely-controlled titrimetry.

The Post Office Engineering Research Station, Dollis Hill, London, indicated the use of compact, low-voltage radiographic equipment. The apparatus shown allowed sensitive radiographic examination of very feebly-absorbing materials such as polythene to be carried out with radiation generated in the range 10-20 kV. Inhomogeneities and inclusions can be located and removed for further study.

A dithizone extraction assembly was exhibited by Birmingham Tame and Rea District Drainage Board. The apparatus can be used for the determination of zinc and cadmium in trade waste samples. It can also be used for determining the synthetic detergents concentration of sewage.

The board also demonstrated a modification of the apparatus described by Wheatland and Lloyd, for determination of the rate of uptake of oxygen by liquids. Its uses include the biochemical oxidation requirements of sewage, trade wastes and the oxygen absorption of gas

liquor in place of the Birate test.

A demonstration was provided by the Water Pollution Research Laboratory, Stevenage, Herts, of a polarographic dissolved oxygen recorder. In this recorder polarographic currents, reproducible over long periods and of about 10 times the usual value, are given by a wide-bore dropping mercury electrode (0.8 mm. internal diameter) pointed upwards at 45°. Direct recording of dissolved oxygen is made possible by automatic compen-

sation for the effect of temperature on the polarographic current.

Since the use of this apparatus was reported in *The Analyst* (1958, 83, 304) a modification has been made in recording by use of a chopper-bar galvanometer-type recorder. Also it has been shown necessary to effect silicone treatment of the dropping mercury electrode to prevent the water seeping back into the jet, wetting the surface.

AEA (Research Group), Royal Arsenal, London, showed the use of low level counting equipment. For determining fall-out strontium 90 and for other biological purposes two types of β -anticoincidence counter were demonstrated. The eight-channel minimum background equipment has a counting efficiency (for >0.5 MeV β -radiation) of approximately 30 per cent with a background rate as low as 0.5 counts per minute. This has been achieved by the use of low background GM4 Geiger counters screened by a suitable geometrical array of brassbodied GE21 Geiger tubes, the whole enclosed within 4 in. of steel shielding. Known as Type 1067A Coincidence Units, they are employed to supply anti-coincidence and total counts from each GM4 to three-channel scalers. Each channel consists of a simple decade scale (EIT) which operates a four-figure register. Slots have been provided in the front and rear faces of the steel assembly to receive Perspex carriers for easy and reproducible positioning of the sample travs.

The other apparatus, automatic β -assay equipment Type 1475A, is standard equipment used by the Atomic Energy Authority for automatic counting of low activity β -sources. A small three-position turntable rotates within the lead castle beneath an anti-coincidence Geiger assembly. Samples are loaded by sliding out the turntable mechanism through a slit in the castle. Background counting rates are approximately 1 count per minute for low-background GM4 and 2 counts per minute for EAM 2S Geiger tubes with an efficiency of 20 to 25 per cent (for >0.5 MeV β -radiation).

Application of dithizone as indicator in titrations was demonstrated by the

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Research and Development Department, Distillers Co. Ltd., Epsom, Surrey. In particular, the titration of sulphate, cyanide and halide ions using dithizone as indicator, with applications to the analysis of organic compounds was described.

Mr. M. S. J. Dallas, Research Department, Unilever Ltd., Port Sunlight, Cheshire, described a method for the determination of sulphur (10 p.p.m.) in oils. This method is based on the reductive desulphurisation of sulphur compounds by nickel catalysts in the presence of hydrogen. A 3 gm. sample containing 10 p.p.m., can be assayed in three to four hours with an accuracy of about ±3 per cent, it is stated.

Advantage of the method lies in the fact that, since nickel catalyst will absorb most sulphur compounds strongly, traces of sulphur can be picked up by a comparatively small amount of catalyst, which can then be filtered off from the oil; this overcomes the difficult oxidation of macro amounts of oils. The adsorbed sulphur compound is quantitatively reduced to nickel sulphide in hydrogen at 150° to 200°C; then the hydrogen sulphide, generated from the nickel sulphide by action of mineral acid, is finally estimated spectrophotometrically after conversion to methylene blue.

Same Reaction Vessel

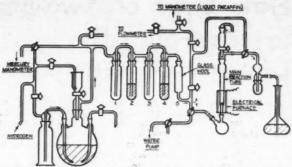
The whole procedure is carried out in the same reaction vessel, which consists of a vertical glass tube with a small sintered disc half way up, and an electric heating jacket on the outside. Arrangement is made for supplying a controlled amount of purified hydrogen to the reaction tube, care being taken to remove traces of sulphur compounds and of oxygen from the hydrogen (by bubbling through NaOH/pyrogallol and through mercuric chloride solutions).

The sample of oil is hydrogenated at 150° to 200°C in the presence of about 50 mg. nickel in the reaction tube, a stream of hydrogen being forced up through the sintered glass disc. Suction is then applied beneath the sintered disc to filter the hydrogenation mixture. The nickel catalyst left on the disc is subsequently heated to 400° to 500°C in a slow downward stream of hydrogen and, when cool, the nickel is dissolved in hydrochloric acid, while an upward stream of hydrogen through the disc carries the generated H₂S into the absorption flask, which contains zinc acetate solution. The zinc sulphide formed is finally converted to methylene blue according to the procedure detailed by Johnson and Nishita (Anal. Chem., 1952, 24, 736).

Applications of this method are: routine estimation of sulphur in oils or fats; determination of traces of a known sulphur compound in liquids of low volatility; and, following the course of isolation of traces of sulphur compounds from biological material.

As some sulphur compounds cannot be quantitatively converted to H₂S, the method in its present form is not applicable to the total assay of sulphur in all continued forms. Compounds which can be determined quantitatively are: sulphur,

Apparatus for determining sulphur in oils, shown by Mr. Dallas of Unilever



p-chloro-thiophenol, a-thiophenic acid, dibenzyl disulphide, phenyl thiocarbamide, diphenyl sulphone, methyl-p toluene sulphonate and potassium sulphate. Low results are stated to be obtained with traces of sulphamic acid and sulphanilic acid. Sulphonal cannot be estimated at all. For any given sulphur compound, however, the results are reproducible under fixed conditions and this has made the method useful in the particular applications mentioned above. Neither nitrogen nor halogens appear to interfere in trace amounts, but large amounts of heavy metals or halogens will completely poison the nickel catalyst.

For determination of alkylimino groups, Atomic Weapons Research Establishment, Aldermaston, Berks, have developed a modified apparatus in which taps or joints are eliminated, and the condensed hydriodic acid distilled from

the reaction bulb during the high temperature distillation is returned to it by a syphon arrangement affording, it is reported, a quick and simple procedure.

reported, a quick and simple procedure. Another apparatus exhibited by AWRE was for the micro determination of nitrogen in organic compounds. It is a modification of the method described in The Analyst (1954). Electrolytic combustion is used for the combustion permitting an analysis to be carried out in 17 to 20 minutes with an accuracy of ± 0.2 per cent.

Preparation of electrolytic silver wool for use in the determination of sulphur by Ingram's empty tube combustion technique was demonstrated by 'Shell' Research Ltd., Thornton Research Centre, Chester. Titration of the silver sulphate formed, washed off the silver wool, is carried out potentiometrically with standard potassium iodide.

Isotope Irradiation Units for Chemical Industry Shown at Geneva

CHEMICAL irradiation units for storing suitable isotopes and controlling their gamma ray emissions for industrial and laboratory research purposes are being manufactured by Nuclear Engineering Ltd., Woolwich Road, London, SE17. One model was among the firm's many exhibits at the Atoms for Peace exhibition at Geneva.

Preliminary research has suggested that many chemical processes may be triggered by irradiation techniques; the polymerisation of monomers and the modification of preformed monomers; the grafting together of substances that do not normally interact; the chlorination of benzene, toluene, and chlorobenzene; alkylation; organic synthesis; the production of ammonia and fixation of nitrogen, and other catalytic effects.

The catalytic or 'initiating' effect is said to remain constant throughout the reaction, whereas a chemical catalyst may be progressively destroyed and thereby cause a falling-off in reaction rate; the effect of radiation is largely temperature-independent; there is no contamination of the product by catalyst residues; the method is applicable to gas, liquid, or solid-phase polymerisations; radiation may bring about polymerisation of unreactive monomers which are not susceptible to the action of catalysts.

Whether irradiation techniques can be

made safe as well as efficient under industrial production conditions has yet to be proved, process by process, and it is to facilitate the carrying out of research programmes that Nuclear Engineering have designed a wide range of storage and handling equipment.



The Patatron CR.500, a twin-source unit for use with 2,000 Curies of Cobalt 60

Flow Laws of Two-phase Systems Met By **Dimensional Analysis**

GENERAL law governing the flow of a two-phase system, cor-responding to the Navier-Stokes equations for single-phase flow, has not yet been established, but recent French work on this subject (1, 2) has led to a dimensional analysis approach to the problem and a new method of representing experimental and theoretical results.

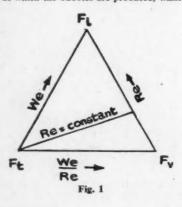
In the case where the gaseous phase is dispersed in the form of bubbles in an otherwise continuous liquid phase, it appears that, among the large number of variables which may be considered, five have a major influence on the flow. These are: d, the diameter of the gas bubbles; v, the velocity of the bubbles relative to the liquid phase; p, the density of the liquid phase; μ , the viscosity of the liquid phase; and σ , the surface tension of the liquid phase.

If the acceleration due to gravity g is added to the five variables above, the usual methods of dimensional analysis give the three dimensionless products, Reynolds Number Re, Weber Number We and Froude Number Fr. If a coordinate system with axes representing Re, We and Fr is prepared, the relation between these quantities may be represented by a surface.

It is easy to show that the only dimensionless product of the three terms Re, We and Fr which does not contain g, but which does contain a characteristic of the liquid phase, is of the form Fr We⁻³ Re⁴; the variable v disappears from all expressions of the form Wep Fre Re-2(p+g) while the variable d is excluded from all expressions of the form Rem Wen Frm+n, thus leading to the above result and the surface which may be constructed in the given coordinate system.

For laminar flow conditions, the equilibrium condition between the pressure forces and the surface tension forces may be written

[(We/Fr) = 6(D/d)]where D is the diameter of the orifice at which the bubbles are produced, while



Stokes equation gives Re = 18 Fr. The intersection of the two planes thus defined is found in the neighbourhood of the surface: Fr We-3 Re4 = constant (for a given physical state of the liquid phase).

For values of Re greater than 2,

An appraisal of some recent French work on the dynamics of two-phase flow in which the relationship between the Reynolds, Weber and Froude numbers is represented graphically in a tetrahedron

by P. G. MORGAN

Lecturer in Engineering, Manchester University

careful measurements by Peebles and Garber (3) give the relation

 $[v = 0.33 g^{0.76} (\rho/\mu)^{0.52} (d/2)^{1.28}]$ This result may be put into the form Re^{2.68} = 2900 Fr^{3.64} corresponding to a physical law of the form Re2 = C Fr3 (c being a constant). This appears to be an interesting continuation of Stokes law, i.e. that Re = 18 Fr.

For the turbulent flow region, experimental results may equally well be expressed as relations between We, Fr and Re, but the coincidence of the surfaces thus determined with the surface of state is less exact due to the effect of other forces, particularly those connected with the gaseous phase.

Further consideration of the dynamics of two-phase systems has shown that when three or four types of force have a predominant influence, an analogy may be considered with the methods of representation used in physical chemistry.

The influence of three types of force may be represented by a triangle, in which each vertex represents one of the forces. Each side of the triangle thus represents the variation of a dimensionless coefficient, defined as the quotient of the two forces corresponding to the points at the ends of that side. At the inside of the triangle, the same value of this dimensionless coefficient lies on the line joining the point to the opposite vertex. For example, if the three forces considered are the inertia force Fi, the viscous force F_v and the surface tension force Ft, then the side Fi Fr represents the variation of Reynolds number, and along the line joining point F_t to any point on the line F₁ F_v the Reynolds Number remains constant (Fig. 1).

The further development of this idea leads, in the case of four types of force,

to a regular tetrahedron, in which the forces represent the four points. This is directly applicable to the study of a gaseous phase dispersed in bubble form in a liquid phase, in which the pre-dominant forces are F_i, F_v and F_t for the liquid phase, together with a gravity force F_g . The three sides F_v , F_i , F_tF_i and F_gF_i represent linearly the variation

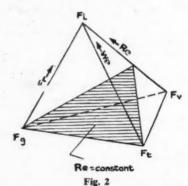
of the three functions $[1+(1/Re)]^{-1}$, $[1+(1/We)]^{-1}$, $[1+(1/Fr)]^{-1}$ respectively, i.e. on hyperbolic scales the variation of Re, We and Fr. (Fig. 2.)

For the interior of the tetrahedron defined above, the regions corresponding to constant values of the Reynolds number are the different positions of planes rotating about the line FtFg. while for the constant Froude Weber numbers the planes pivot about the lines F_vF_t and F_vF_g respectively. Each point within the tetrahedron corresponds to a value of Re, We and Fr, approaching zero in the plane FeFvFt

and infinity towards the point F_i.

The relationship between Re, We and Fr, determined theoretically or by experiment, may be represented in the tetrahedron or in one of its sections. In particular, the surface given by $Fr We^{-3} Re^4 = F_i^2 F_t^{-3} F_v^{-4} F_g^{-1} = constant$, which has been discussed previously, may be represented.

Incidentally, this graphical representation is equally applicable to a wave on



the surface of a liquid, where the forces Fi, Ft, Fg, and, to a lesser extent, Fv, predominate, and it is probable that other phenomena may be similarly represented.

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 3. Peeblea, F. N., Garber, H. J. Chem. Eng. Progress, 1953, 49, 88.

Ashburton Chemicals Parkland Site for Factory

Planning permission has now been granted to Ashburton Chemical Works, of Old Trafford, Manchester, a sub-sidiary of Geigy (Holdings), for the building of a chemical factory on a 100acre site forming part of Glynllifon Park, near Caernarvon (see CHEMICAL AGE, 17 May, p. 909).

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A VALVE in Fluon introduced by the Chemical Pipe & Vessel Co. Ltd., God-

stone Road, Kenley, Surrey, can be used with hot corrosive liquids such as strong sulphuric acid, strong nitric acid and hot ferric chloride. In fact, it is claimed to be immune to corrosion by any known chemical. It is workable up to 200°C, a much higher temperature than is possible with rubber-lined valves.

These valves, made from ½ to 2 in. bore, are available with flanged ends, and in some cases screwed ends for screwing into low pressure lines. The range includes fine adjustment valves, pressure relief valves, on- and off-valves, and non-return valves. Price of the 1 in. valve is about £35.

HIGH-TOROUE LABORATORY STIRRER

A HIGH-TORQUE variable speed electric stirrer that can conveniently be clamped

on a retort stand is supplied for laboratory use by Griffin and George (Sales) Ltd., Ealing Road, Alperton, Wembley, Middlesex, at £22 10s.

Specially designed for dealing with the more viscous liquids such as paints, oils



and varnishes, the Griffin geared stirrer incorporates a 3:1 reduction gear and built-in resistance to allow continuously variable control over the speed range 300-1,300 r.p.m. at 6-7 oz./in. torque.

DIAPHRAGM **PRESSURE** METER

A NEW mercuryless meter to measure a range of differential pressures from 20 in.

to 200 in. of water at static pressures up to 2,000 p.s.i. has been introduced by Foxboro-Yoxall Ltd., Redhill, Surrey. Advanced design features include a frictionless flexure-pivoted drive unit of exceptional sensitivity, positive protection against overrange with no zero shift, a simple method of range change, a built-in temperature compensation for all differential ranges, and full range damping which is adjustable under pressure.

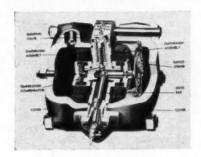
A flexible diaphragm assembly is used for the measurement of differential pressure. Each preformed disc is welded

EQUIPMENT REVIEW

Chemical Plant: Laboratory Apparatus: Safety and Anti-corrosion Products

and shaped to nest into the adjacent disc and then welded to a spacing ring. Excess pressure in either chamber compresses the diaphragm assembly until each pair of discs is nested and the spacing rings have contacted to form a metal-to-metal stop.

This assembly, it is claimed, has greater flexibility and higher sensitivity



Mercuryless diaphragm meter

per unit length with less spring rate than spun or hydraulically-formed bellows for equivalent service.

The drive unit is of solid rods joined by a metal flexure and forms a direct connection between the diaphragm assembly and the pen lever. The special range spring, the temperature compensator and the damping valve are exclusive Foxboro features and make the new Type 37 an exceptionally reliable and accurate mercuryless meter.

FOR PIPED **URANIUM ORE**

A GAMMA RAY electrometer has been developed by Electronic Instruments Ltd. in conjunction with the UK AEA, to meet

a special need for measuring the concentration of uranium ore in slurry flowing through steel pipes in ore treatment plants. The absorption of the gamma rays in a pipe varies with the density of the slurry. This change in absorption, however, is small compared with the total gamma ray flux incident on the ionization chamber. An instrument was therefore required that would measure a small change in the presence of a large signal.

This electrometer is designed around the EIL Vibron unit, an electro-mechanical device which replaces the electrometer valve and provides a far higher degree of sensitivity and stability than can be attained by conventional valve circuits. Manufacturers are Electronic Instruments Ltd., Lower Mortlake Road, Richmond, Surrey.

NEW **MULTI-POINT** RECORDER

A MULTI - POINT recorder which provides a permanent, printed

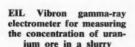
record of variable inputs (strain, temperature, d.c. supply, etc.) from any one of 500 points, the time at which the measurement was taken and the channel identification, is being produced by Savage and Parsons Ltd., Watford, Herts. A prototype model arranged for measuring temperature was shown on the company's stand at the 'Atoms for Peace' exhibition in Geneva.

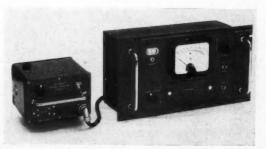
The recorder consists of a main unit and from one to ten extension units coupled to it through a junction box. The main unit contains the printing mechanism mechanically coupled to a motor-driven slide-wire potentiometer, a high gain amplifier for the control of the motor and servo system, a precision voltage supply for comparing with thermocouple and other d.c. inputs, and associated power supplies. Three types of extension units are now availableone for strain work, one for thermocouple inputs and one for straightforward

VENTILATED PROTECTIVE TIUZ

A PROTECTIVE suit made of Geon p.v.c. sheeting has recently been introduced

jointly by Spembly Ltd., Manor Road, Chatham, Kent, and Plysu Products Ltd., Woburn Sands, Bletchley, Bucks. Designed and developed primarily to meet the requirements of the UK Atomic Energy Authority, this suit is said to





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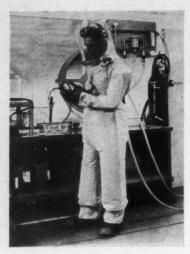
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Spembly pressure ventilated suit

afford the wearer complete protection against radio-active dust while allowing maximum freedom of movement.

The Spembly suit is also useful for the inspection of petroleum tanks and gas holders, refrigeration plant emergencies or wherever there may be atmospheric contamination by toxic fumes, gases or dust particles. Special features include full ventilation to the body extremities, and helmet air supply, both controlled by the wearer; reduced fatigue since the weight of the air hose is taken by a special harness; and unrestricted vision in all directions.

CORROSION RESISTANT WELLGLASS

For use in oil refineries, chemical plant and similar situations, A.E.I. Lamp

Lighting Company Ltd. are marketing a new 150/200 w. vapour-proof tungsten wellglass fitting. This has a corrosionresistant LM-6-M aluminium alloy canopy, tapped for top entry (1 in. electrical thread), and a heat resisting wellglass protected by a galvanised wire guard. There is a 14 in. diameter p.v.c. reflector available as an optional extra and the fitting can also be supplied with either a 45 deg. angle entry or conventional side entry.

The wellglass is held in position by the galvanised wire guard by means of a screwed pad with brass wing screw fastening which exerts pressure on the wellglass and "Neoprene" sealing gasket. The unit is supplied complete with lampholder to take 150 or 200 w.

lamps with E.S. caps.

temperatures just below acid dew-point as determined by the conductivity method; the peak of maximum sulphuric acid corrosion would appear to be some 30° to 40°C below this acid dew-point, the quantity of acid condensed is at a maximum corresponding to the peak of corrosion and the acid so produced is more dilute and more aggressive than that produced at acid dew-point; increased water vapour content of flue gases increases the rate of acid condensation. If flue gases are at temperatures of the

order of 500°C and come in contact with a metal surface below acid dew-point then corrosive attack will take place. This has been noticed particularly in hot water boilers when metal temperatures are of the order of 80° to 90°C while gas temperatures may be from 300° to 800°C. It is suggested, moreover, that although air heater design usually arranges for turbulent gas flow over metal surfaces the thermal conductivity of the system is such that the metal surface exposed to the flue gases remains below dew-point at least until the combustion air has reached a temperature of the order of 100°C. An extremely thin boundary film is therefore likely on the flue gas side of the metal and in this film acid mist occurs which condenses on the metal.

During trials with Teramin it was noted that the amine preparation had caused considerable coagulation of starch solids which were deposited in various parts of the air heater and inside the tubes. These deposits were moist and highly acidic and contained very small quantities of soluble iron. In no case were the deposits bonded and simple water washing was all that was found to be necessary before recommissioning.

Teramin attacks copper and cuprous alloys, but tin plating or zinc galvanising and monel metal are not affected. Neoprene has been found suitable as a sealing material on sight glasses and plain asbestos string or soft lead are also useful joining materials, since Teramin dissolves most organic materials.

Heterocyclic Tertiary Amines as Corrosion Inhibitors

I JSE of amines and compounds thereof as 'restrainers' or corrosion inhibitors in acid pickling baths has been practised for many years as a means of removing scale without attacking the underlying metal. The use of these substances for prevention of corrosion by flue gas, however, has been a new conception.

Research carried out by Midland Tar Distillers Ltd. indicated that the introduction of some 0.03 per cent to 0.05 per cent of tertiary amines by weight of fuel into flue gases caused a reduction in the corrosion of mild steel probes by some 80 per cent. Further experiments were carried out by BCURA using Teramin, Midland Tar's trade name for these amines, and further probe trials were arranged in collaboration with OPD department of Shell Petroleum Co. Ltd. In all cases the tests substantiated the original trial.

Teramin is a derivative of the distillation of coal tar. The various grades of the product are all inflammable liquids of viscosity similar to that of kerosene. The boiling range may be adjusted to suit particular applications, but normally Teramin 2 is supplied with a boiling range from approximately 160° to 200°C Flash point of this grade is about 100°F. It is insoluble in water but soluble in dilute acids and most organic solvents. Specific gravity lies between 0.980 and

The amines are inflammable and it is not feasible to add the inhibitor to the fuel or to add it to the products of combustion at a point in a furnace system where combustion could take place. Thermal stability has been examined by experiments carried out under rigorous conditions. The tertiary amines used for corrosion inhibition can be considered to be thermally stable at all temperatures involved in their application. At higher temperatures and in the absence of air, higher boiling point material is cracked to give pyridine, picolines and other less complex tertiary amines. The grade of Teramin selected should not be totally evaporated, the company states.

Several theories have been proposed regarding the actual mechanism whereby tertiary amines prevent acid attack on iron. It is suggested that in so far as acid attack on ferrous metal is accompanied by the production of surface electric currents, the acid acting as an electrolyte, then inhibition of corrosion may be due to Teramin producing a polarisation

Rate of attack of flue gases on mild steel plotted against the temperature of the mild steel surface shows that two well defined corrosion zones occur, the first is that associated with the temperature at which sulphurous acid solution in water condenses on the metal and the second that corresponding to sulphuric acid condensation.

It has been found that: given a sulphur content greater than 1 per cent by weight on the fuel, sulphuric acid of some 70 to 80 per cent concentration will appear on metal surfaces if these are at

Metal-Chelating Compounds
A new catalogue of Judex and Judacton analytical reagents and other laboratory chemicals is available from the General Chemical and Pharmaceutical Co. Ltd., Judex Works, Sudbury, Wem-New sections include blev. Middx. specifications of more than 200 of the more popular Judex analytical reagents AR; an increased range of special reagents, including metal indicators; a list of materials suitable for use in chromatographic techniques; and a range of some of the newer metal chelating compounds.

The range of metal-chelating compounds comprises: diaminocyclohexanetetra-acetic acid: 2:2'-diaminodiethyl-ether tetra-acetic acid; diaminoethane-NN'-di-(o-hydroxyphenyl)acetic acid; diethylene triaminepenta-acetic sodium salt, solution, about 33 per cent NN-di-(2-hydroxyethyl)-glycine, sodium salt, solution about 33 per cent w/v; N-(hyroxyethyl)-diaminoetranetriacetic acid, sodium salt, solution, about 33 per cent w/v; iminodiacetic acid; nitrilotriacetic acid.

Overseas News

DEGUSSA ACHIEVE NEW SYNTHESIS OF HCN FROM METHANE AND AMMONIA

DEGUSSA of West Germany have recently completed a technical synthesis of prussic acid from methane and ammonia without the addition of oxygen. This new synthesis was realised by Degussa in a plant with a capacity of 100 ton/month of hydrocyanic acid.

The BMA process (Blausaure aus Methan und Ammoniak) is based on large-scale utilisation of the original ingredients-90 per cent use of methane and 80-85 per cent use of ammonia-and on obtaining a high concentration of prussic acid in the resultant gas (up to 23 Vol per cent). The dry gas remaining after the extraction of the prussic acid consists of 96.2 per cent hydrogen, which may be looked on as a by-product of the process. The synthesis is carried out at temperatures of from 1,200° to 1,400°C in a new furnace which utilises sintered alumina reaction tubes. The furnace has eight chambers, each holding a vertical bundle of 13 alumina tubes. Six gas burners in each chamber provide the 100 k.cal./mole needed to sustain the reaction. The tubes, 6.5 ft. long and 0.8 in. dia., are coated inside with a thin layer of platinum-based (70 per cent) catalyst.

With the usual method of producing hydrocyanic acid—by the Andrussov method—the utilisation of methane is about 70 per cent and that of ammonia about 60 per cent. The hydrocyanic acid concentration in the product gas amounts to 6 to 7 per cent.

US Borax To Increase Boric Acid Output

Production of boric acid at the Wilmington, California, plant of US Borax and Chemical Corporation is to be increased to meet greater demand from many industries and to supply the immediate needs of boron fuel manufacturers. The company has recently been awarded two contracts by Olin Mathieson Chemical and Callery Chemicals, producers of boron fuel.

Acetylene Factory to be Set up in Beira, PEA

A factory for the preparation of oxygen, acetylene and nitrogen gases is to be set up in Beira, Portuguese East Africa, states Barclays Bank DCO. Estimated output will be 18,000 cu. m. of oxygen and 9,000 cu. m. of acetylene per month. The site for the new factory is at present being cleared for building operations. This project is being financed entirely by Portuguese capital.

Sasol Plant to Increase Output by 30 per cent

South African Coal and Gas Corporation (Sasol) announce that they have begun work on a vast consolidation scheme, which is expected to raise the value of production by 35 per cent within a year of its completion. Erection of additional standby equipment is planned. At present it is necessary to close down the existing unit periodically for maintenance and inspection.

Standby equipment now being built includes boilers and synthesis reactors. Cost of the extension scheme will be about £4 million. It will be finance either by sales proceeds or by short-term loans. Completion will take two years.

ENI Plan New Fertiliser Factory In Sicily

The Italian ENI group plan next year to invest a large sum in prospecting in Sicily for potassium. Once concessions are obtained, ENI will build in the island a plant for the production of potassic fertilisers which will supplement the nitrogenous fertiliser plant that they are operating at Ravenna. In view of the expanding output of petroleum at the oil field operated by ENI at Gela, Sicily, the group says that it will build on the site a unit for the production of petrochemicals.

Greek Fertiliser Plant To Be 'Made In Germany'

On the advice of a Danish firm of consulting engineers, the Greek Government have given the contract for the construction of the projected \$35 million (£12½ million) nitrogen fertiliser plant at Ptolemais to the German 'Uhde' and the Italo-Swiss 'Ammonia Casale' groups. About 80 per cent of the plant equipment will, it is reported, be made in Germany.

The new plant should make Greece self-sufficient in nitrogen fertilisers, resulting in a saving of about \$15 million (£5 million) in foreign exchange.

No Protection For Indian Para-Aminosalicylic Industry

The Indian Government has accepted inter alia the recommendation of the Tariff Commission that there is no case at present for granting protection to the p-aminosalicylic acid industry, whether by way of exemption of duty on m-aminophenol or protective tariffs or subsidy.

The Government has also accepted the recommendation that protection to the Indian bichromates industry should be discontinued from 1 January next.

Spanish Company to Manufacture Diphenyl

It is announced that Electro-Química de Flix, SA, of Barcelona, owned by Cros-Kønzern and Farbwerke Hoechst AG, are to make 400 tons of diphenyl at their works. The diphenyl produced, it is stated, will be used by them for manufacture of chlorinated derivatives.

Australians Invent New Uranium Extraction Process

A new Australian process for extracting uranium ores, known as the pulp-in-resin technique developed by the Industrial Chemistry Division of CSIRO, is being tested at Rum Jungle, and may be used in the treatment of ore from a new uranium desposit there. The method involves the passage of a pulp of uranium and other minerals through a bed of resin from which the metal may be extracted by chemical solvents.

US Courtaulds Have New Cross-linked Cellulosic Fibres

Two new fibres, Topel and Corval, have been developed by Courtaulds (Alabama, US). They belong to a new family of cross-linked cellulosic films, having distinctly different properties from rayon, acetate or cotton.

The following properties, which are associated more with Corval than Topel are: comparable water absorption and swelling to those of cotton (the fibres can be yarn-dyed in 100 per cent form); good alkali resistance and so can be mercerised in blends with other synthetic fibres; the fibres can be resincoated with a wide range of cross-linking material; and, can be dyed with all dyes normally used on cellulose fibres.

dyes normally used on cellulose fibres. Corval is being recommended for blending with synthetic fibres and wool, to improve consumer properties. Price for commercial quantities is 40 cents per lb. in bright and dull, regular and crimped, and in all standard deniers up to 15. Topel is for use alone or blended with cotton, acetate or nylon. Particular good blends with cotton can be obtained owing to its relatively low elongation at break and high alkali resistance, Courtaulds claim. Price of Topel is 34 cents a lb. in small commercial quantities. Only an uncrimped form is available in 1½ and 3 denier in both bright and dull.

One-man Formaldehyde Plant To Produce 5,000 Tons A Year

A fully automatic formaldehyde plant capable of producing 5,000 tons a year has been completed by Reichhold Chemie AG in Hamburg, the first RCI plant outside the US to make this chemical. The whole production process is centrally controlled by one service man.

US Engineering Firm Sponsors International Symposium

A symposium of licensees of the Scientific Design Co.'s 125 patents controlling the direct air oxidation process of producing ethylene oxide, was held at the Linden, New Jersey, plant of the General Aniline and Fibre Corporation earlier this month and attracted delegates from France, Germany, Japan and the US.

It is believed that this was the first international meeting ever sponsored by an

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engineering firm to bring together in confidential sessions process licensees with a common interest. Data from full-scale plant operation was made available by many delegates on a reciprocal basis.

BASF 'Go Slow' on Fertiliser Extensions

Badische Anilin- und Sodafabrik, Ludwigshafen, have started to use a base other than coal for the production of much of the 'Sythesegas' used in the manufacture of ammonia compounds. Coal is no longer economical, says the company.

BASF also announce that during 1957, they produced more ammonia compounds and artificial fertilisers than in 1956, without working at full capacity. The company had declined to share in the building of several plants for the manufacture of fertilisers, since it believed that the industry was growing faster than the demand.

Para Xylol Plant For Japan

The Mitsui Petrochemical Industries Ltd., of Tokyo, are to erect a p-xylol plant with a capacity of 5,000 tons a year at Iwakuni, near Hiroshima. The plant, to be completed by next Summer, will by using two established processes under licence produce pure p-xylol out of a usual xylol mixture. By the Octafining process, it will convert o-xylol, m-xylol and ethylbenzole to p-xylol by means of a catalyst. These two processes are patents of Standard Oil Co. (Indiana) and Atlantic Refining Co. respectively.

China Is Stepping Up Fertiliser Production

A modern phosphorus ore dressing plant with an annual capacity of 300,000 tons of concentrated ore has gone into production at Nanking, and two phosphate fertiliser plants—one at Nanking and the other at Taiyuan—with a total capacity of 600,000 tons, are on stream. These were designed by Chinese engineers with Soviet assistance.

A new fertiliser plant is being built in Yunnan province with an annual capacity of 100,000 tons of synthetic ammonia, 200,000 tons of double super-phosphate and 100,0000 tons of fused calcium magnesium phosphate.

Other chemical industry developments under way in China include the mass production of 67 of 137 scheduled new dyestuffs—including indanthren and sulphur dyes, and dyes with an active radical—at Shanghai, and an agrochemical plant now under construction at Taiyuan, Shansi province, designed to produce 40,000 tons of high polymer insecticide and plant hormones annually.

Chicago Institute's New Hydrogasification Process

A method of converting Colorado oil shale into high quality fuel gas was described at the recent national meeting of the American Chemical Society in a report by two chemical engineers of the Institute of Gas Technology, Chicago. The method is called hydrogasification, and used to produce gas with a heating value of 800 B.Th.U. per cu. ft., economically and efficiently, according to Eugene B. Schultz, who presented the report.

Preliminary calculations are stated to show that the process is more efficient than shale-to-oil conversion, since it makes use of 90 to 100 per cent of the organic matter in the shale instead of about 80 per cent. Economic aspects of the hydrogasification process appear to be promising. It is estimated that the finished gas will cost only 30 cents per thousand cu. ft., based on the cost of mining and crushing shale containing 30 gal. of crude oil per ton. It is a one-step process involving the treatment of crushed shale with hydrogen at high temperature and pressure.

Recent figures show about 1,260,000 million barrels of oil contained in the Colorado oil shale deposits. This compares with an estimated 250,000 million barrels expected from domestic crude oil recovery.

German Lime Firms Seek a Syndicate

The West German lime manufacturing cartel, Kalkkontor Rheine, has applied to the German Government for permission to form a syndicate. This would have the effect, says the cartel, of reducing the price of lime products, including artificial manures, in Western Germany since, with a common marketing system for the six firms which form the cartel, marketing expenses would fall considerably.

The four member companies of Kalkkontor Rheine are: Kalkwerke Otto Brekweg GmbH; Rheine GmbH; Rheiner Bau- und Düngekalkwerke; Nette and Co. KG; Wettinger Kalkwerk H. Engels and Co.; and Kalkwerk Wettringen and Schencking and Co. GmbH.

US Output of Sulphuric Acid Below 1957 Level

Output and consumption of sulphuric acid in the US so far this year are below comparable figures for 1956 and 1957. Acid demands differ considerably for different areas in the country; operating rates in the west and south-west having declined very little, while on the east coast consumption has shown the greatest drop.

Gross production of sulphuric acid in the US (100 per cent basis) in the period January-April 1958 was 5,225,837 tons

(5,608,207 tons in January-April 1957).

Frasch sulphur production and consumption in the US in recent months have been relatively steady, but at lower levels than last year. Recovered sulphur, which now makes up about 10 per cent of US domestic elemental sulphur availability, has shown a steady growth in both output and use.

Portugal Aims To Export Nitrogen

Annual production of ammonium sulphate by the Estarreja factory of Amoniaco Portuges SARL has been raised to 70,000 tons since additional equipment supplied from West Germany has been installed at a cost of Es240 million (£3 million). This is a part of a Es1,500 million (£19 million) scheme to raise production of nitrogenous fertilisers to 350,000 tons annually, providing for increased home consumption and an export surplus.

New German Plant to Produce Pure Propylene

A plant for the production of pure propylene is now being built at the oil refinery of the German DEA concern at Heide. The propylene will be based on propane produced at the Heide cracking plant. Like the ethylene at present produced by DEA, the propylene will be used as a base for petrochemical products.

Lubricants with Excellent Radiation Stabilisation

Monsanto Chemicals and Shell Development in the US have developed for the US Air Force, a series of radiation-resistant polyphenyl ethers having two to five times the radiation stability of present organic lubricants. The compounds were investigated for possible use as lubricants, reactor coolants, or hydraulic fluid. Tests are now being applied in full scale equipment under combined heat and intense radiation. It has been found that the compounds retain their lubricating properties throughout the liquid range—20° to 800°F and show no thermal decomposition below 800°F.

Russian Polyorganometallic Siloxanes

THERE is much interest in the Soviet Union's silicon production which has been built up by Professor Adrianov of the Academy of Sciences, Moscow. Two new types have been exhibited at the Brussels Exhibition.

The first new group resembles silicones in molecular structure, but contain in the basic molecule a metal atom such as aluminium, nickel or cobalt. The general formula is (-O-RSiR-O-Me-O-RSi-R-O-)n. In the aluminium compounds the third valency of aluminium is satisfied by an -O-SiR₃ group. Cobalt compounds form violet coloured resins and the nickel, green coloured resins, R can be aliphatic or aromatic. Co-polymers can also be made. According to the Russian workers, the resins will resist

temperatures of up to 500°C. This resistance to high temperatures is believed to be due to the presence of the metal atoms. Condensation polymerisation occurs under heat without the need of a catalyst. The degree of condensation can be varied at will and hence the molecular weight.

The resins have been reinforced with asbestos, glass fibres, synthetic fibres and also natural fibres. Numerous compounds can be produced, it is reported, and some of these plastics materials are apparently being used in building missiles.

The second group of organo-metallic siloxane resins are stated to be similar to the first group. They have the general formula [-O-Al(O-SiR₃)-]_n where R is either aliphatic or aromatic.

- MR. WESLEY WICKERSHAM, vice president in charge of operations for Allied Chemical International, is touring Europe to investigate the European market and the technological advances made by European chemical companies. He is visiting France, Belgium, Holland, West Germany, Great Britain, Switzerland, Italy and Spain before returning to his headquarters in New York City in October.
- MR. BASIL W. BALLS, technical sales manager of Foxboro-Yoxall Ltd., Redhill, is in the US for consultations with the Foxboro Co. on new products, with particular reference to the new ECI range of electronic control instruments.
- MR. WILLIAM H. MCFADZEAN, chairman and managing director of British Insulated Callender's Cables Ltd., and other companies in the BICC group, has been elected deputy president of the Federation of British Industries.
- THE DUKE OF EDINBURGH has agreed to become patron of the congress of the International Federation of Associations of Textile Chemists and Colourists to be



continental subsidiaries, has joined the board to advise on merchandising and continental matters. Mr. G. C. D'Arcy Biss, the third new director, is a senior partner in a firm of solicitors.

MR. W. M. BROWNLIE, B.Sc., head of the veterinary science division of the research department, Boots Pure Drug Co. Ltd., and MR. E. W. G. CROUCH, N.D.A.,



At the summer school in dyeing at Nottingham University, organised by the Society of Dyers and Colourists—lecturers Dr. W. R. Moore (left) and Dr. R. L. Elliott (right) both of the Bradford Institute of Technology, with Mr. A. W. Carpenter, chairman of the organising committee

held at Church House, London, on 16-18 September 1959. The congress is being organised by the Society of Dyers and Colourists.

- Directors of Fisons (Canada) Ltd., the new company that will be responsible for the development, promotion and marketing in Canada of the agricultural chemicals of Fisons Pest Control Ltd. and J. R. Geigy, SA, Basle, include: MR. A. WORMALD, commercial director of Fisons Ltd., and chairman of Fison Pest Control Ltd., MR. W. ABEL-SMITH, Fisons Ltd., and PROFESSOR A. W. A. BROWN, University of Western Ontario. The new company's agricultural chemical department will start trading on 1 October from 1893 Davenport Road, Toronto 9.
- Three new appointments to the board of Aspro-Nicholas are announced, MR. R. G. SMITH, director of the Australian associated company for eight years, is coming to England to be director in charge of research and development and MR. J. CHALIER, a director of several

agricultural marketing manager of the company's international division, are visiting African territories to gain first-hand information on the effectiveness of Boots products on bovine trypanosomiasis. Boots produce a prophylactic and a treatment for this disease.

■ MR. MORTON GORDON has been appointed acting chief welding engineer of Metal and Pipeline Endurance Ltd., Artillery House, London SW1, in succession to Mr. R. B. WHALLEY, who has resigned.

- MR. OLIVER GREGORY, export sales manager of Griffin and George (Sales) Ltd., laboratory furnishers, Ealing Road, Alperton, Middlesex, who earlier this year visited the middle-European countries, is now touring northern Europe, calling on the group's agents in Germany, Denmark, Finland, Sweden and Norway.
- MR. GEORGE WATSON, general manager, opened the recent annual conference of representatives of the Crookes Laboratories, held in London with a review of the previous financial year. MR. J. R. BOWDEN, sales director, was in the chair. DR. N. O. Eve spoke on the clinical uses for corticotrophin (ACTH) and MR. N. TAYLOR on 'the commercial importance of piglet anaemia'.
- SIR MILES THOMAS, chairman of Monsanto Chemicals Ltd., is chairman of a new development corporation for Wales and Monmouthshire that has been set up with headquarters in Cardiff by a group of industrialists who have large interests in the principality.
- MR. G. L. TOWERS, B.Sc., has joined Flexibox Ltd., Nash Road, Manchester 17, as sales engineer. Based on Birmingham, he will be responsible for sales and service in the Midlands area.
- MR. C. I. RUTHERFORD, who has joined the board of the ICI Fibres Division, has been division production manager for two years.

Wills

COLONEL FREDERICK BLYTHE late chairman and managing director of William Blythe and Co. Ltd., Holland Bank Chemical Works, Church, Lanes, chairman and managing director of John Riley and Sons Ltd., chemical manufacturers, Hapton, Burnley, and a director of Sizing Materials Co. Ltd., chemical exporters, Manchester and London SWI, who died on 26 July 1957, left £285,619 net.

SIR JAMES SWINBURNE, F.R.S., a pioneer in the development of plastics and chairman of Bakelite Ltd., who died on 30 March, aged 100 years, left £43,291 net.

Geon P.V.C. used for BP Acid Effluents

Acid effluents at BP's Kent refinery are handled by 11,000 ft. of 4 in. and 6 in. bore Hipact rigid p.v.c. piping manufactured by Extrudex Ltd., Bracknell, Berks, from Geon RA 170 high-impact p.v.c.



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Commercial News

Ashe Chemical Ltd.

Shareholders in Ashe Chemical Ltd. have adopted a new article which empowers the board between 15 September and 1 October next to sell at current

market prices holdings of less than 50 shares for the benefit of those holders.

Mr. F. T. Wright, chairman, reports the receipt of proxies from 499 share-holders representing 2,439,393 shares in favour of the resolution, and from 23 shareholders representing 7,143 shares against. Holders of less than 50 shares will be asked before 26 September if they want to retain their shares.

Aspro Nicholas

An unchanged interim dividend of 6 per cent is declared by Aspro-Nicholas Ltd. A total of 24 per cent was paid for the year to 31 March 1958. It has also been announced that trading results for the first three months ended 30 June 1958 continued to be satisfactory.

Beecham Group

The Beecham Group, which recently made a successful bid for the ordinary capital of Thomas and Evans, has declared a first interim of 7 per cent for the year ending 31 March next on the £10.2 million ordinary, as doubled by a scrip issue. The new shares to be issued under the offer do not rank for this interim.

British Petroleum

Group sales for the half-year ended 30 June 1958 of British Petroleum Co. increased by £17.8 million to £404.2 million. The company are proposing to capitalise £101.2 million of reserves in making a one-for-one scrip issue.

Interim dividend on account of 1958 is maintained on the existing capital at 8 per cent tax free. The final dividend will be on the increased capital. Holders are informed, however, that it should not be inferred from the proposed capitalisation that the dividend for the current year will be greater than for 1957.

The half-year's net income was £25.9 million (£22.4 million in the first half of

Burt, Boulton and Haywood

The new phthalic anhydride plant of Société Chimique de Selzaete, Belgium, in which Burt, Boulton and Haywood hold a majority interest, is now almost complete and production is expected to start shortly. This was stated by Captain F. J. E. China, chairman, in his report, presented at the recent annual meeting of Burt, Boulton and Haywood.

Calico Printers

Group profits of the Calico Printers Association for the year ended 30 June were £2.65 million (£2.95 million). Net profit was £601,116 (£750,598), with the parent company's net profit at £592,094 (£752,769). Royalty income, after tax,

• Belgian Phthalic Plant Nears Completion

• CPA Terylene Revenue Up, but Profits Down

Yorks. Imperial Metals Bid for Tube Firm

Lawes Cut Dividend on Lower Profits

from Terylene and other patents was £300,000 (£247,000), the net increase being due to further expansion of Terylene sales by ICI and Canadian Industries and of polyester fibres by European manufacturers.

Total distribution is maintained at 22½ per cent, with the dividend reduced from 15 to 10 per cent and the bonus increased 5 per cent to 124 per cent. The increased bonus is intended to recognise the special nature of the additional revenue from royalties but, having regard to the lower profit on trading activities, it is felt prudent to recommend a lower payment on ordinary.

The directors add that no decision has yet been reached in connection with the application for prolongation of the UK patents for Terylene, which is being opposed by British Celanese.

Horace Cory

Alterations to the articles of Horace Cory and Co., chemical colour manufacturers, are being recommended by the directors so as to provide that any distribution upon a capitalisation of reserves shall be for the exclusive benefit of the ordinary shareholders. The fixed preference dividend is to be increased to 61 per cent and the existing participation right will remain unchanged. The preference dividend reserve fund will be increased to £3,250.

Subject to the approval of these proposals the directors have further decided to recommend the capitalisation of £20,000 of the general reserve by the issue of four new ordinary 1s shares for every one held.

The Distillers Company

The directors of the Distillers Co. Ltd. have declared a dividend on the preference capital for the six months ending 30 September of 3 per cent, payable on 15

Hudson and Wright

Yorkshire Imperial Metals, formed earlier this year by ICI Metals Division and Yorkshire Copper Works, have made an offer of 6s. 9d. per share for all the 2.21 million 2s. ordinary shares of Hudson and Wright, manufacturers of seamless brass and copper tubes. The directors of Hudson and Wright have recommended acceptance of the offer.

Lawes Chemical

Group net profit of Lawes Chemical for year ended 30 June was £58,451 (£65,887). Dividend of 14 per cent (12½ per cent) has been declared. Annual meeting will be held at the May Fair Hotel, London W, at 12.30 p.m. on 6 October.

The chairman states that the setback to profits was due partly to absorbing cost increases and partly to heavier expenditure on research and on publicity. Lower current costs of materials and freights are now being passed on in the form of reduced prices to customers and, in conjunction with the company's sales campaign, should strengthen its position in the agricultural fertiliser market.

Nuclear Chemical Plant

The new British consortium that plans to produce and process nuclear fuels and chemica's for export (see CHEMICAL AGE, 6 September, p. 397) has now been registered as Nuclear Chemical Plant Ltd., a company with £99 authorised capital. First directors are Robert W. Rutherford, M.I.Chem.E., joint deputy managing director of Power-Gas Corporation Ltd. (capital £1½ million); George G. Farthing, a director of Humphreys and Glasgow Ltd. (capital £400,000); and John H. N. Thompson, deputy chairman John Thompson Ltd. (capital £2,400,000). The registered office is at Barrington House, Gresham Street, London EC2.

The declared objects of the new company are 'to carry on the business of designers and dealers in plant, machinery, tools, implements and accessories of all kinds for the production, manufacture, treatment, processing, storage, handling and transport of nuclear chemicals and

C. A. Parsons

Scientific instrument, machinery, etc., manufacturers, C. A. Parsons and Co., are maintaining their interim dividend at 33 per cent on the £9 million ordinary capital in respect of 1958. A similar final payment made a total of 7½ per cent for

I. G. Farben

Assets abroad of I.G. Farben were given an estimated value of DM 1,203 million (£102 million) last week by Dr. W. Schmidt, one of the liquidators of the former German chemical combine. Schmidt thought that DM 1,091 million (£93.2 million) could be definitely considered as lost. He said that continued existence of I.G. Farben was legally necessary to enable the reacquisition of the nationalised assets in East Germany, once a unified German state was restored. The company's activity will, it is expected, be reduced considerably within the next two or three years because most of the liquidation would by then be complete.

Lonza AG

The 61st annual report of Lonza AG, one of Switzerland's largest chemical (Continued in next page)

ACCIDENT RATE IN CHEMICALS IS BELOW UK AVERAGE

Safer Working with Fluorine Compounds

THE chemical industry is among the most safety-conscious of British industries, for the accident figures for 1957 recorded in the annual report of the chief inspector of factories show a reassuring trend. The frequency rate—calculated as the number of lost-time accidents per 100,000 man-hours worked—at 1.25 is well below the national average of 1.61, and shows an improvement over the previous year of 6 per cent compared with the national improvement of 5 per cent.

The new Chief Inspector of Factories, Mr. T. W. McCulloch, in his report for 1957 (HMSO, 5s), points out that the use—or misuse—of machinery is not the major cause of accidents in workshop and factory. Far more people are hurt handling goods carelessly, and by falling. Gravity is still the greatest menace.

The report gives examples of dangerous industrial chemicals that during the past year were being replaced by others of a safer nature.

Perchlorethylene was replacing white spirit and trichlorethylene in the dry cleaning industry, steam replacing benzene for the extraction of fat from bones; ethyl alcohol was replacing methyl alcohol, while methylated spirit was being increasingly used instead of isopropyl alcohol for foundry mould-facing compounds.

Liquid Sulphur Cuts Risks

By using liquid sulphur instead of dry sulphur in certain processes, risks of explosion were reduced, while plastics materials were replacing inflammable celluloid in paper cutting machines. Toluene was gradually replacing benzene for softening plastic tubes.

The use of organic fluorine compounds as grease solvents also promises to improve the safety figures in the future. Hitherto, Mr. McCulloch points out, grease solvents seemed to be either inflammable and non-toxic, or toxic and non-inflammable. Fluorinated hydrocarbons now being manufactured on a small scale offer good solvent properties, and some are both non-toxic and noninflammable. Although these are at present scarce and expensive, the cost should decrease with greater demand, and then a safe grease solvent will be available to replace the toxic chlorinated hydrocarbons and the inflammable petroleum fractions now widely used.

Further fields in which the fluorinated hydrocarbons are being developed are in the control of fire, where trifluorobromomethane has shown excellent extinguishing properties (its vapour has proved to be virtually non-toxic), and as refrigerating liquids in refrigerators.

The human factor in the drive towards maximum safety is still one of the major problems. There is little difficulty, for instance, in designing respirators capable of filtering out fine dust particles; the problem is still to persuade workers to wear them, because inefficient equipment that only deals with larger particles visible to the naked eye is more attractive in appearance.

The growing use of radioactive isotopes in industrial processes is posing a serious problem to the factory inspectorate, since no one can foretell how rapidly the technique may spread or how serious may be the menace to health both in the factories and out.

The possibility of a careless worker discharging significant quantities of radio-

active waste into the public sewers or refuse-collecting systems is very real; and although at the end of 1957 only about 400 factories were using radioactive isotopes (and 85 per cent of them are using sealed sources of radiation) a new army of Harwell-trained factory inspectors is being recruited with a view to maintaining a satisfactory level of control. The strength of this force at present is just under 40.

It is probable, says the report, that the hazard to the health of workers is greatest where sealed sources of radiation are being used for radiography and fluoroscopy. There is little precise information about the amount of radiation to which workers using sealed sources are exposed. This is because there is no code of special regulations as a system of control, although a draft code was published in 1957.

Legislation enforcing regulations now being finalised in committee is expected to pass through Parliament early in 1959.

Commercial News

(Continued from previous page)

firms, states that total turnover for the year to 31 March showed an increase of 10 per cent over the previous 12 months. Some 250,000 tons of products were sold, of which 17½ per cent—representing half the total value—were new products developed by the company's research and development laboratories. Net profit increased slightly from Sw.Frs. 5.3 million (£434,000) to Sw.Frs. 5.6 million (£459,000) and the dividend remained unchanged at 7 per cent.

INCREASES OF CAPITAL

AEROSOL ASSOCIATED INDUSTRIES LTD., valve manufacturers, etc., Crewe House, Curzon Street, London W1. Increased by £49,900 beyond the registered capital of £100.

M. L. ALKAN LTD., chemical manufacturers, 3/4 Clements Inn, Strand, London WC2. Increased by £80,000 beyond the registered capital of £20,000.

ASPRO-NICHOLAS LTD., 16 Berkeley Street, London W1. Increased by £500,000 beyond the registered capital of £1,500,000.

COALITE AND CHEMICAL PRODUCTS LTD., Buttermilk Lane, Bolsover, nr. Chesterfield. Increased by £2,000,000 beyond the registered capital of £2,000,000.

HYDRONIL SYNDICATE LTD., chemical manufacturers, 14 Gloucester Road, London SW7. Increased by £8,400 beyond the registered capital of £7,000.

NORGINE PHARMACEUTICAL PRODUCTS (LONDON) LTD., 26/8 Bedford Row, London WC1. Increased by £10.000 beyond the registered capital of £12,000.

PFIZER LTD., chemical manufacturers, ets., 137/9 Sandgate Road, Folkestone, Kent. Increased by £1,000,000 beyond the registered capital of £2,000,000.

WESTMINSTER LABORATORIES LTD., manufacturing chemists, etc., Chalcot Road, London NW1. Increased by £50,000 beyond the registered capital of £150,000

Chemical Stocks & Shares

Markets Remain Quiet

STOCK markets over the last two weeks have had a quiet period, the main influence being the unsettled Far Eastern situation. There has, however, been no general setback in prices. With the Government's recent declaration that there will not be a general election this autumn, investors have had time to reconsider their programme.

The general feeling suggests that equities are not over priced; indeed, some people may come round to the view that they are somewhat under priced. A gradual rise in chemical equities, however, is likely to continue to reflect the weight of demand and one can expect a reasonable capital appreciation at today's purchase prices.

	1958			Change
High	Low	Security	15 Sept.	week
18/10	13/48	Albright & W. 5/-	17/101	+3d.
20/71	16/9	Bakelite 10/-	19/9	_
20/41	14/103	Borax 5/- Dfd.	17/-	_
15/6	10/44	Bt. Glues 4/-	15/44	-
6/63	6/18	B. I. Plastics 2/-	6/6	+21d.
44/9	28/3	Bt. Xylonite	44/-	+3d.
54/9	45/9	Fisons	54/6	+71d.
44/-	31/6	Glaxo 10/-	43/9	_
28/9	21/9	Hickson & W. 10/-	28/9	+3d.
34/15	24/3	ICI	33/9	- I d.
3/41	2/74	Kleeman I/-	3/11	-
18/6	14/-	Laporte 10/-	18/3	+6d.
16/21	12/6	Monsanto 5/-	14/9	_
15/6	10/103	Reichhold 5/-	15/3	$\pm 1/3$

ICI's New Terephthalic Acid Plant is by Amoco

IT IS Now known that the new process to be used under licence by ICI to make terephthalic acid at the rate of 30 million lb. a year at Wilton (see CHEMICAL AGE, 23 August, p. 294) is the US Amoco oxidation process. Three other such plants are in operation or under construction—Amoco's own at Joliet, Illinois, and two in Japan.

ICI's Terylene expansion programme,

ICI's Terylene expansion programme, scheduled to go on stream in 1960, will boost the Wilton output to 50 million

lb. a year.

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NEW PATENTS

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Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

AMENDED SPECIFICATIONS

On Sale 8 October or as soon as possible thereafter

Synthesis of octyl alcohols. Standard Oil De 663 240 velopment Co. Nickel electro deposition. Harshaw Chemical Co

ACCEPTANCES

Open to public inspection 22 October

Method for producing zirconium metal. National Lead Co. [Addition to 758 763.] 803 356 1-Ethynyl-cyclohexyl ethyl carbonates. Dow Chemical Co.

Process for producing peracids from aliphatic carboxylic acids. Union Carbide Corp. 803 159

Open to public inspection 29 October

Production of cellular synthetic rubbers. Xetal Products, Ltd. 803 771 ystemic herbicides compositions. British Rubber Producers' Research Assoc. (Baptist, E. D. C., and Jonge, P. de) 863 772 and Jonge, P. de)

rocess and installation for separating and storing tar. Still, K. F. [trading as Still, C. [firm] ofll Producing roducing nodu'ar cast iron. Union Ca Corp. [Divided out of 803,657, 803,652]

803 651 Process of intercalcating olefins into methanes. Farbwerke Hoechst AG Manufacture of amides. Ciba Ltd. 803 464 Alkylation of hydrocarbons utilising evaporative cooling. Stratford Engineering Corp. Preparation of silicon tetrafluoride. Grace & Co. W. R. Dehydrocortical hormones. Upjohn Co. 803 655 Dehydration process. Celanese Corporation America.

Process for producing an aleurone extract from cereal seeds. Aleuron S.p.A. 803 664
Basic dyestuffs. Farbenfabriken Bayer A.G. 803 525 materials. Imperial Chemic Photographic

Metabolic processes. Imperial Chemical Industries, Ltd., Borrow, A., Jefferys, E. G., and Nixon, I. S. 803 591 NIXON, 1. S.
Polyamides and their production. Beck, Koller & Co. (England), Ltd.
Southetic Resin Compositions. Beck, Koller & Co. (England), Ltd.
803 518

Dyestuffs of the anthraquinone series. Imperia Chemical Industries, Ltd. 803 473 Polymeric materials comprising low pressure poly olefines. Petrochemicals, Ltd. [Cognate appli

cation 13 074.1 803 557 Cation 13 U/4.]
Filtering medium for use in brewing beverages.
Spicelman, P. P.
Process for purifying hydrocarbons, especially olefines. Ziegler, K.
Sedimentation tanks. Hartley, C. J.
803 558
803 598

Gas-liquid contact apparatus. Koch Engineer
Co., Inc. 803 711

Dioxane pretreatment of polyester structures Dioxane pretreatment or polyester structure:
Chemstrand Corp.

803 77

Fuel oil composition containing alkyl amine salts
Socony Mobil Oil Co., Inc.

803 47

Treatment of gases and vapours containing hydre
gen sulphide. Bahr, E. E. 803 774 803 474

803 475 Linear polyamides. British Nylon Spinners, Ltd.

Pouring spouts for liquid containers. Dimond.
B. T. J.

Catalytic reforming of hydrocarbons and appara-tus therefor. Gas Machinery Co. 803 788 Production of finely-divided silicon dioxide. New Jersey Zinc Co. on of a leucocytosis factor. Nyegaard

Co. A.S. 803 775 Esters of chloramphenicol. Boehringer & Soehne

Separation by extractive distillation of syclopen-tane from neohexane. Bataafsche Petroleum Maatschappij N.V., de. 803 763 De-rusting and de-oxidising preparations. Tilhaud,

803 498 R. Manufacture of unsaturated ketones. Hoffmann-La Roche & Co. A.G., F. 803 765 Apparatus and process for carrying out reactions which proceed endothermically in fluidised layers. Badische Anilin- & Soda-Fabrik A.G.

Production of I-ketocyclo-octyl-2-methylene com-pounds. Badische Anilin- & Soda-Fabrik A.G.

Manufacture of hydrogen. Esso Research

Manutacture of hydrogen. Esso Research & Bog 766
Production of alkaline-earth metals. Farbenfabriken Bayer A.G. Polymeric basic aluminium salts of organic acids. Koninklijke Industrieele Maatschappij Vorheen Noury & Van der Lande N.V. 803 768 Process for separating gaseous or vaporous sub-stances more especially isotopes. Deutsche Gold- und Silber-Scheideanstalt Vorm. Roessler. Deutsche

Steroids and the manufacture thereof.

Production of N-hydrocarbon substituted amino Production or N-nydrocarbon substituted amino-carboxylic acids, their esters or salts. Badische Anilin- & Soda-Fabrik A.G. 863 778. Utilisation of phosphoric acid in the preparation of greases. Esso Research & Engineering Co.

Acid inhibitors. Technion Research & Develop

Acid inhibitors. Technion Research & Development Foundation, Ltd., Heimann, H., Kohn, D. H., and Israel, Y.

Protecting foodstuffs from fungal attack. United States Borax & Chemical Corp. 903 692. Method of removing very small particles suppended in a gas, and apparatus therefor Leuder, H.

Process and cetalist for collections with the second state of the second s

Leuder, H. 803 543
Process and catalyst for polymerisation of polymerisable hydrocarbons. Phillips Petroleu

Coating compositions. Du Pont de Nemours Co., E. I. [Addition to 763 158.] 803 780 Preparation of 2-ethyl-2-sec-butyl-1, 3-propanediodicarbamate. Carter Products, Inc. 803 477 803 780 rocess for producing alkanolamides of α, γ dihydroxy-β, β-dimethyl butyric acid. Ferrosan

AR 803 769 Solubilising of mineral, vegetable, animal oils for cosmetic, pharmactutical and industrial pur-

cosmetic, pharmactutical and industrial purposes. Lubowe, I. I.

803 545
Manufacture of nitriles. Downes, H. I. (Batasf-sche Petroleum Maatschappij N.V., de) 803 478
Process and bath for descaling metals. Du Pont de Nemours & Co., E. I.

Vinylic type organosilcon compounds.

Silicones, Ltd.

803 478
Midland \$803 479
Acid description of creaties by decreasiles.

Acid decomposition of organic hydroperoxides. Edogawa Kagaku Kogyo Kabushiki Kaisha

Dehydrocortical hormones. Upjohn Co. [Divided out of 803 655.] [Addition to 761 526.] 803 656 Aqueous solutions of salts of di-(4-amidinophenyl)-triazene-(N-1: 3) and process for preparing them. Farbwerke Hoechst AG 803 750

Market Reports

EXPORT CALL IS LESS ACTIVE

LONDON Reports indicate that the movement in chemicals during the past week has been of reasonable dimensions, with the home industrial outlets taking fair quantities against contracts. flow of new business has been steady although buyers are hesitant to enter into commitments beyond their immediate needs. The call for shipment is perhaps a little less active. Prices generally remain at recent levels with the undertone keeping firm.

A steady home demand for creosote oil, cresylic acid, and pitch has been noted among coal-tar products, but market conditions are quiet with conditions unchanged.

MANCHESTER Apart mainly from the textile and allied industries, contracts for heavy chemical products are being drawn against fairly well by most of the leading industrial consumers in Lancashire and West Riding. Replacement buying, however, leaves room for improvement on both the home and export sides, and a number of users are committing themselves for shorter delivery periods than normally. A moderate aggregate tonnage of fertiliser materials is being taken up, with a quietly steady demand reported in the tar products section.

GLASGOW There has not been a great deal of activity during the past week in the Scottish market. Most transactions were for the usual range of industrial chemicals, and the quantities involved were nominally against current requirements. Prices showed little change. The export market is showing reasonable interest while the position of agricultural chemicals is still quiet.

DIARY DATES

TUESDAY, 23 SEPTEMBER Soc. of Instrument Technology—Birmin 7 p.m. Visit to Midland Tar Distillers, Ltd.

WEDNESDAY, 24 SEPTEMBER
OCCA & SDC—London: Royal Soc. of Tropical
Medicine and Hygiene, 26 Portland Place. 7 p.m.
'Surface activity: the general concept & some
consequences' by Dr. J. L. Moilliet.
Inst. of Pet. & Armer. Soc. for Testing Materials
—London: Senate House, Univ. of London, Gower
Street. Conference on mass spectrometry. Until
26 September.
Plastics Inst.—Leeds: St. Marks House 184

20 September.

Plastics Inst.—Leeds: St. Marks House, 186
Woodhouse Lane. ICI paper on 'Polyurethane foams'.

foams'.
Industrial Fuel Efficiency Exhibition—London:
National Hall, Olympia. 10.30 a.m. to 6 p.m.
Inst. of Fuel—London, Olympia. 10.30 a.m. to
12.30 p.m. Technical meetings on better fuel
utilisation. Also on 26 & 30 September &
October.
SCI—London: 14 Belgrave Squire, SWI. 6.30 p.m.
'Living polymers' by Prof. M. Szwarc.

'Living polymers' THURSDAY, 25 SEPTEMBER
Textile Inst.—Manchester: 10 Blackfriars Street.
7 p.m. 'Use of tirchlorethylene in continuous scouring & bleaching of textiles' by W. A. S.

FRIDAY, 26 SEPTEMBER
Inst. Metal Finishing—Sheffield: Grand Hotel.
7 p.m. "Surface preparation & porosity in electrodeposited nickel" by P. A. Brook.
Polarographic Soc.—Ardeer: ICI Nobel Division.
One-day symposium.

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TRADE NOTES

Microscope Exhibition

An exhibition of microscopes and other precision optical instruments will be held at the works of W. Watson and Sons Ltd., West End Lane, Barnet, Herts, on 23, 24 and 25 September. A wide range of microscopes, many set up for demonstration, will be on show.

CJB Moves to New Offices
Constructors John Brown Ltd. have moved to their new offices at CJB House, Eastborne Terrace, Paddington, London W2 (Ambassador 8080). From 20 September this address and telephone number should be used, except for the following departments: purchasing, progressing and shipping department, which remains at Duncan House, Dolphin Square, London SW1 (Victoria 3571) until 1 Decemter, when it will be moved to CJB House; automatic control division and instrumentation workshops, which will remain at Roxby Place, Seagrave Road, London SW6 (Fulham 7761) until further notice; and the plant department which will remain at Cold Blow Lane, London SE14 (New Cross 0267) until further notice.

Agents for Italian GR-S

Synthetic rubbers produced by ANIC, the Italian government-controlled petrochemical plant in the Po valley, are to be distributed in the UK and most European countries by Lewis and Peat

Ltd., (Rubber) Plantation Mincing Lane, London EC3. The range of rubbers at present available include GR-S 1500, 1502, oil-modified 1712 and cable grade 1503.

It is claimed that because these polymer products are made from natural gas and are therefore independent of crude petroleum supplies, prices are highly competitive.

Whiffen and Sons Ltd.

The executive and sales organisations of Whiffen and Sons Ltd. are now concentrated at the Loughborough factory, Willows Works, Derby Road, Loughborough, Leicestershire, although it will still be possible to contact members of the sales organisation through Fison House. The publicity and advertising department will remain in London at Fison House, 95 Wigmore Street, London W1.

BP Detergents Ltd.

New name adopted for Irano Products Ltd., the BP company formed in 1945 to distribute By-Prox and Comprox detergents, and, who more recently have marketed paraffin wax and paraffin coke, is BP Detergents Ltd.

Mixer Business Acquired

The business of Campbell and Co. (Manchester) Ltd., manufacturers of mixers and agitators, has been acquired

by Autodrome (Engineers) Ltd., Shepley Works, Audenshaw, nr. Manchester, The company has been reorganised under the title of Campbell (Manchester Sales) Ltd., and is now operating from Ajax Works. Campbell Street, Walkden. In addition to the standard range of mixers, the company is offering a more comprehensive service of design and fabrication to specialised needs, with vessels or other process equipment where required. The range of basic units is from 1 h.p. to 250 h.p. and 1 to 1,500 r.p.m.

Changes of Address

From 22 September the sales office of Foamite Ltd. will be at Clifton House, 83 Euston Road, London NW1, tel. Euston 5578.

To meet increasing demand and to accommodate an expanding programme, British Arca Regulators Ltd. are moving on 6 October from Millbank, London SW1, to Sisson Road, Gloucester, where all administrative functions and production of light engineering products will be carried out. Manufacture of heavy equipment will be continued at the works at Ledsam Street, Birmingham.

Chemico's Address

The London address of the Chemical Construction (Gt. Britain) Ltd. was given as 'Bush House, Aldwych, London WC2' in their advertisement which appeared in our issue of 13 September. The correct address should have been 'Henrietta House, Henrietta Place,

FIRST SAFETY

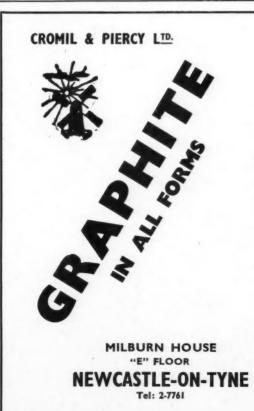
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ANALYTICAL METHODS

A series of post-graduate lectures on more recent analytical techniques will be held during the Autumn Term on Tuesdays at 7 p.m., commencing on 7th October, 1958. The topics will include spectroscopic methods, solvent extraction, square wave polarography, radio-activation, isotopic dilution, inorganic paper chromatography, silicate analysis, nuclear magnetic resonance and organic sub-micro analysis. A leaflet giving full details and an application form may be obtained from the Head of the Chemistry Department, Chelsea College of Science and Technology, Manresa Raad London S W 3 Road, London, S.W.3.

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